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—OF THE—

American Ceramic Society

A Monthly Journal devoted to the Arts and Sciences Related to the Silicate Industries

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JOURNAL
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A monthly journal devoted to the arts and sciences related to the silicate industries.

Vol. 3 **January, 1920** **No. 1**

EDITORIALS

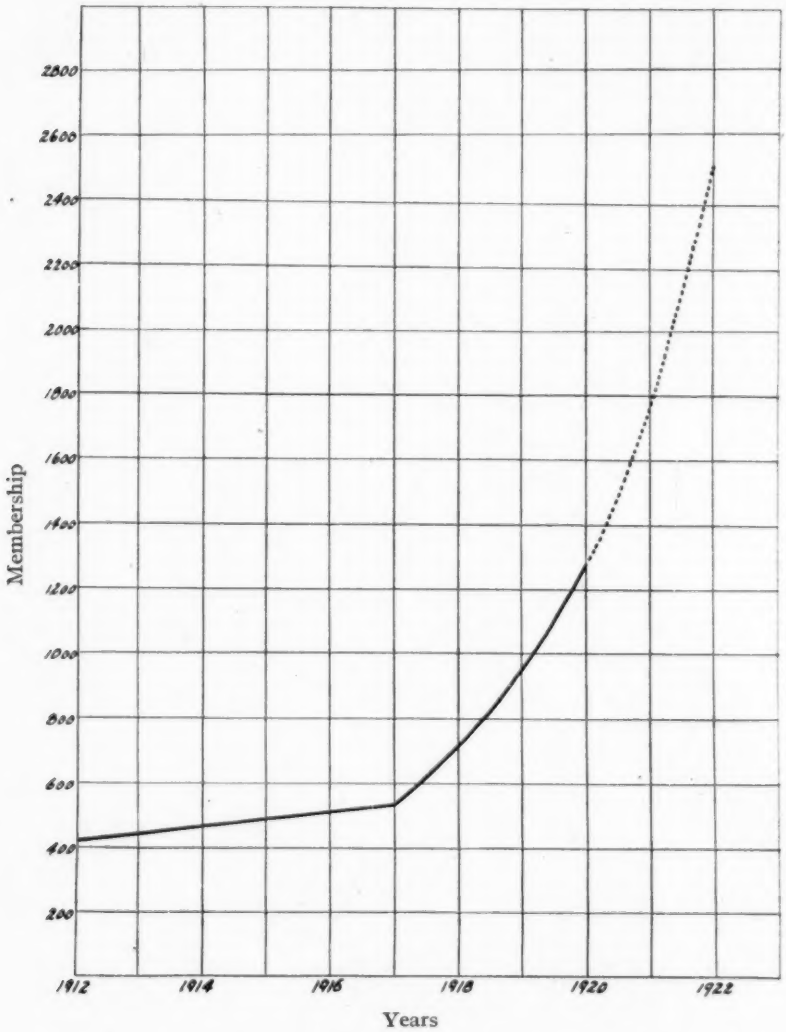
MINIMUM OF 2500 MEMBERS BY JANUARY 1, 1922

The accompanying graph shows the growth in membership of the American Ceramic Society during the past eight years. It will be seen that the growth from 1912 to 1917 corresponds with the slow rate of increase in membership during the first eighteen years of the life of the Society. With the annual meeting in 1917, came the reorganization of the Society and energetic prosecution of activities along a diversity of lines, including the launching of the Journal. From that time on growth has been rapid.

A noticeable and very important feature of the growth during the last three years is that the total increase each year has been greater than that of the preceding. During 1917, 216 new members were gained; in 1918, 226; and in 1919, 284. This results in a concave curve when the total membership is shown in graph form. This concave form of the curve is very encouraging, since it shows that we are gaining momentum. It also places a greater responsibility on the present membership if the form of the graph is to be maintained, for it means that a rapidly increasing number of members must be obtained each year. It means that we must gain 540 new members during 1920 and 700 during 1921. In other words, we must secure approximately twice as many members during the present year as we did last year and three times as many next year.

The growth in membership that we expect during the next two years cannot be obtained merely by solicitation. *It must be earned.* It will not come merely by strenuous activity of the mem-

bership committee, although such activity will be necessary. It must come as the result of energetic and effective work by every



officer, every committee and every member to make this Society of greater service to the ceramic industry. By officers and com-

mittees we mean not only those of the general Society, but also those of the various Divisions and Sections.

The field for obtaining new members is ample. There are over 250,000 people engaged in the ceramic industries of this country. If we attain to 2500 members by the date set we will have less than one member for each 100 persons engaged in these industries. If the Society becomes the great technical organization that it should, and that we intend to make it, the ratio of members to persons in the industries should be much higher than this. Twenty-five hundred members by January 1, 1922, is to be considered as the permissible minimum consistent with healthy and normal growth of the Society and not as the maximum.

Does it not seem then that each of us should take stock of our possible abilities, not our abilities when we are lazy and indifferent, but when we throw ourselves whole-heartedly into an effort to attain some goal? Let each of us do the one thing that lies in our power to make the Society as efficient and serviceable as possible. If we do this the American Ceramic Society will deserve at least 2500 members by January 1, 1922, and *it will get them.*

A CALL FOR LEADERS

At present our membership in certain branches of the ceramic industry, such as the cement, the lime, and the brick and tile, is small. The activities of the Society in behalf of these branches and the amount of service we are giving to these industries should be increased. The work of the Society in other directions, also, can well be expanded. We have the facilities and the ability to do this. All that is lacking is leaders with enough vision to perceive the opportunities and with sufficient courage to undertake the tasks. That such men will be found is evident by the success the Society has had in the past three years in finding men to conduct its committee work, to organize divisions and to form local sections. The most remarkable and the most stimulating feature of the life of the Society during this period has been the large number of men, especially of comparatively young men, that by right

of efficiency and of devotion to the interests of the Society have earned positions as leaders in the Society's work. Such men are the most valuable asset of the Society at present and its most substantial assurance of growth and development in the future.

If the Society is to maintain its present rapid rate of growth and expand its field of service, it is imperative that leaders be found to conduct its activities along lines and in fields hitherto neglected. These men must be found not in two years from now, nor in one year, but immediately. It is difficult for the Board of Trustees to find these men, for, like gold, ability and devotion is often found in unexpected places. This is no time for undue modesty, and we may feel assured the Board of Trustees will be very glad indeed to have any man who wishes to work for the good of the Society volunteer for service. Place and the opportunity will be found for him.

OUR PLATFORM

1. To make this Journal indispensable; to make it so serviceable that no one in this country who is interested either intellectually or financially in any phase of ceramic technology can afford *not* to be a conscientious reader of the JOURNAL OF THE AMERICAN CERAMIC SOCIETY.

2. To make this Journal the great technologic journal of the ceramic industries of this country, not a trade paper on the one hand nor a scientific publication on the other, but a true technologic journal, to which the contributions of the trained scientist and of the factory man, richly endowed with ideas and data by practical experience, must be sent in order to reach an appreciative audience.

3. To make this Journal a comprehensive and timely abstract journal, one that each month shall contain in its abstract department a summary of the progress during the previous month of the whole world in ceramic technology.

4. To make this Journal an impelling force in the growth and development of the Society, by keeping the membership as a whole informed of the various activities of the Society and by holding our editorial columns open to any one who has a message

that will make the members *think* about the welfare of the Society. We do not care whether his ideas are in accord with our own or not. As long as the members *think* about the welfare of the Society it is bound to prosper.

5. To keep this Journal as far as possible mechanically perfect and rhetorically correct, not that we consider form as important as substance, but because good form in composition adds to the pleasure of reading and is due the dignity of a high-class technical journal.

6. To make this Journal a valuable and well patronized advertising medium. This will be a logical result of the successful accomplishment of the foregoing.

ORIGINAL PAPERS AND DISCUSSIONS

THE USE OF SUBSTITUTES FOR TIN OXIDE IN GLAZES

By R. H. Minton

A search of our *Transactions* reveals that little work has been done with substitutes for tin oxide in glazes, outside of the enameled iron field. In enamels for cast iron and sheet steel the principal substitute for tin oxide has been antimony oxide. A review of the Transactions develops the following references:

J. B. Shaw, in "The Allowable Limits of Variation in the Ingredients of Enamels for Sheet Steel," Vol. XI, p. 151, says: "SnO_2 produces opacity alone, but it may be entirely replaced with ZnO plus Sb_2O_3, with equally good results; 0.13 equivalent of ZnO plus Sb_2O_3 or SnO_2 is sufficient to produce a good white opacity by using two coats of white."

Homer F. Staley, in "The Control of Fusibility in Enamels," Vol. XIII, p. 506, states: "While bone ash can be used as a weak opacifying agent to some extent, it is liable to cause minute pinholes in the enamel and vesicular structure in any part of the piece that should happen to be overheated."

R. E. Brown, in "The Replacement of Tin Oxide by Antimony Oxide in Enamels for Cast Iron," Vol. XIV, p. 752, writes: "The effect of Sb_2O_3 is to increase the maturing temperature, and to increase the whiteness and opacity when employed between the limits of 0.0–0.9 equivalent Sb_2O_3." To produce a good white with SnO_2 0.15 equivalent was required.

Homer F. Staley, "Antimony Compounds as Opacifiers in Enamels," Vol. XVIII, p. 188: "Oxide of antimony enamels of good gloss can be made." "Opaque whites are produced by controlling the heat treatment and composition."

Emley and Young, "The Production, Manufacture and Use of Compounds of Barium," Vol. XVIII, p. 247: "Barium salts are also used extensively in the preparation of mat glazes. An important use of barium is in the manufacture of enamels for iron. When tin oxide is used as an opacifier, the presence of barium is very desirable. . . . . "

Ercill C. Hill, "White Terra Cotta Glazes at Cones 6–7," Vol. XVII, p. 383, found that: "The opacity and white color of the glazes increases with the increase of SnO_2. It also increases with increase of ZnO, and with increase of BaO when the ratio of CaO to BaO is less than 1 : 1. Increase of SnO_2 is more effective in increasing opacity than either ZnO or BaO."

Kirkpatrick, Arnold and Geiger, "An Attempt to Fit Enamels to Plastic Clay Bodies," Vol. XVIII, p. 145, found that 0.12 equivalent of SnO_2 was sufficient to produce opacity in an enamel for bricks.

J. B. Shaw, "Antimony Oxide as an Opacifier in Cast Iron Enamels," *J. Am. Ceram. Soc.*, Vol. I, p. 511, asserts that: "Extreme care in proportioning the raw materials, very careful and thorough mixing, and proper smelting, will insure the successful use of antimony oxide."

Homer F. Staley, "Enamels for Cast Iron," *J. Am. Ceram. Soc.*, Vol. I, p. 706, says that sodium metantimonate has been extensively used in America during recent years as an opacifying agent for cast iron enamels which are leadless, or with low or medium lead. He states that "The essential difference between sodium metantimonate enamels and those in which the oxide of antimony is used as the chief opacifier is that the latter contain much larger amounts of sodium nitrate."

These references show that practically all of the investigations into the matter of substitutes for tin oxide as an opacifier has had to do with enamels for metals. The work done by Hill in the field of terra cotta glazes covers the ground very well, but he tried only ZnO and BaO to determine their effects upon SnO_2.

Scope of this Investigation.—In the work under consideration twelve different substitutes were used to replace tin oxide in four

different types of glazes. The corresponding number in each of the four series contains the same opacifier.

SERIES R.—Raw lead glaze at cone 02

			Batch weights
0.70 PbO			167.7 White lead
.10 K_2O	0.15 Al_2O_3	1.7 SiO_2	55.7 Feldspar
.10 CaO		.10 SnO_2	10.0 Whiting
.10 ZnO			8.1 Zinc oxide
			12.9 China clay
			54.0 Flint
			14.9 Tin oxide

SERIES F.—Fritted wall tile glaze at cone 02

			Batch weights
Frit 0.24 K_2O			133.7 Feldspar
.28 Na_2O	0.24 Al_2O_3	3.56 SiO_2	123.6 Flint
.48 CaO		1.12 B_2O_3	48.0 Whiting
			106.9 Borax
			69.4 Boric acid
Glaze 0.49 PbO		2.25 SiO_2	193.5 Frit
.12 K_2O	0.19 Al_2O_3	.571 B_2O_3	126.4 White lead
.15 Na_2O		.10 SnO_2	18.1 China clay
.24 CaO			19.5 Flint
			14.9 Tin oxide

SERIES T.—Terra cotta raw glaze at cone 6

			Batch weights
0.200 KNaO			111.4 Feldspar
.375 CaO	0.325 Al_2O_3	2.50 SiO_2	37.5 Whiting
.175 BaO		.10 SnO_2	34.5 Barium
.250 ZnO			20.2 Zinc oxide
			32.2 China clay
			63.0 Flint
			14.9 Tin oxide

SERIES P.—Raw porcelain glaze at cone 9

			Batch weights
0.36 K_2O			174.9 Feldspar
.23 ZnO	0.54 Al_2O_3	4.00 SiO_2	30.0 Whiting
.35 CaO		.10 SnO_2	16.2 Zinc oxide
.06 BaO			9.8 $BaCO_3$
			38.7 China clay
			78.0 Flint
			14.9 SnO_2

Substitutes Used for SnO_2.—In the first number of each series 0.10 SnO_2 was used, and in the succeeding numbers the same amount by weight of the substitutes was used to replace the tin oxide. Some of these substitutes are so composed that it is not possible to use them by equivalents, and as they are all purchased by weight, that is the only practical method of comparison when price is considered.

Glaze 1—Tin oxide marketed by Roessler & Hasslacher Chemical Co.
Glaze 2—Terrar from Roessler & Hasslacher Chemical Co.
Glaze 3—Zirconium oxide from A. D. Camp, Cleveland.
Glaze 4—Zirconium oxide from Aluminum Alloys Co., Niagara Falls, N. Y.
Glaze 5—Zirconium oxide (pure) from Pennsylvania Salt Co.
Glaze 6—Fluorspar.
Glaze 7—White antimony oxide.
Glaze 8—Aluminum oxide (hydrated)
Glaze 9—Kryolith, from Roessler & Hasslacher Chemical Co.
Glaze 10—Leukonin, from Roessler & Hasslacher Chemical Co.
Glaze 11—Stannolite, from Wenger's, Ltd., England.
Glaze 12—Phosphorite, from Wenger's, Ltd., England.
Glaze 13—Lithopone, from Harrison Bros., Philadelphia.

Data on Substitutes.—"Terrar" is an imported product largely used in the enamel iron industry before the war made it impossible to obtain it. It is probably composed largely of zirconium oxide and zirconium silicate. "Leukonin" is also used in enamel iron work, and is essentially sodium metantimonate. It is not recommended for general glaze purposes. "Stannolite" and "Phosphorite" are compounds whose composition is unknown. They are said to be used very extensively in England for opacifiers in place of tin oxide. "Lithopone" is a compound of 30 per cent zinc sulphide and 70 per cent barium sulphide. It is now being used very largely by the rubber manufacturers in place of zinc oxide.

Firing.—All glazes were made up in batch weights in grams, thoroughly ground in a ball mill, and applied to small grogged body tiles which had previously been fired to cone 9. Series "R," "F" and "T" were fired in a large test kiln to the indicated heats. Series "P" was fired in a large sanitary kiln requiring 120 hours for firing.

Results from Firings.—

SERIES R.

R- 1—Semi-opaque. Milky white. Not well glossed.
R- 2—Less opaque than 1. Not so white.
R- 3—Not quite so opaque as 2. Bubbled.
R- 4—About the same as 2.
R- 5—Similar to 3. Bubbled.
R- 6—Clear glaze. Crazed.
R- 7—Slight opacity. Full gloss.
R- 8—Clear glaze.
R- 9—Clear glaze. Badly crazed.
R-10—Same opacity as 7.
R-11—Same opacity as 7.
R-12—Clear glaze. Crazed.
R-13—Clear glaze. Not fully matured.

Tin oxide shows the best opacity in this series. Would require at least 0.15 equivalent for good opaque glaze. None of the substitutes offer much encouragement, although "terrar" might show results if used in much larger amounts than is required of the tin oxide.

SERIES F

F- 1—Semi-opaque enamel. Glossy.
F- 2—Slightly more opaque than 2, but blistered.
F- 3—Not quite so opaque as 1. Dull surface.
F- 4—Less opaque than 3. More gloss.
F- 5—Clear glaze. Full gloss.
F- 6—Clear glaze. Full gloss.
F- 7—Slight opacity. Full gloss.
F- 8—Clear glaze. Full gloss.
F- 9—Clear glaze. Full gloss. Crazed.
F-10—Slight opacity. Less than 7. Full gloss.
F-11—Slight opacity. Less than 7. Full gloss.
F-12—Clear glaze. Glossy.
F-13—Same as 11. Crazed.

Tin oxide is the best opacifier in this group. The commercial zirconium oxide in 2 is the next best. The pure zirconium oxide in 5 shows no opacity whatever. It is evident that this type of fritted glaze is not at all suited to use for opaque purposes. Glaze "R" and glaze "F" are about equal.

SERIES T

T- 1—Good opaque enamel. Glossy.
T- 2—Fair opaque enamel. Glossy. Not quite so white as 1.

T- 3—Good white opaque. Beaded. Not quite so white as 1.
T- 4—Fair opacity, but less than 2. Glossy.
T- 5—Good white opaque. Not quite equal to 1.
T- 6—Only slight opacity. Crazed.
T- 7—Opaque white. Almost equal to 1 except that it is bubbled.
T- 8—Fair opaque white. Mat surface.
T- 9—Slightly more opacity than 6. Semi-glossy.
T-10—Good opacity. Glossy. Not fully freed from bubbles.
T-11—Semi-opaque. Glossy.
T-12—Semi-opaque. Glossy.
T-13—Semi-opaque. Slight gloss.

There are more opaque glazes in this series than in the first two. Zirconium oxide (pure) in 5 seems equal to tin oxide for opacity, but the glaze is not quite so white. Antimony oxide is equal to tin oxide for opacity but the glaze is not fully developed. Aluminum oxide produces a very good opaque mat glaze. Leukonin seems almost equal to tin oxide for opacity, color and gloss.

SERIES P

P- 1—Good opaque white. Crawled some.
P- 2—Semi-opaque white. Same as "T"-2.
P- 3—Same opacity as 1. Not so white. Crawled.
P- 4—Same as 2.
P- 5—About the same as 3. Not quite so good as 1.
P- 6—Almost a clear glaze.
P- 7—Flocculent opaque white. Crawled.
P- 8—Semi-opaque. Less than 2 and 4.
P- 9—Clear glaze. Glossy.
P-10—Slight opacity. Glossy.
P-11—Semi-opaque; less so than 8.
P-12—Slight opacity. Glossy.
P-13—Slightly opaque, material in suspension.

Tin oxide still seems to be the best opacifier, although "terrar" and zirconium oxide, especially the pure, seem to offer possibilities of being good substitutes. More material might be required for the same opacity as from tin oxide, but the other defects can no doubt be overcome.

General Conclusions

1. Zinc oxide is of great assistance in producing opacity with tin oxide and with the substitutes, as is shown in series "T" and "P."

2. Barium exerts a marked influence on opacity, as shown in a comparison of series "T" with series "P."

3. Zinc and barium oxide have more influence on opacity than the clay content.

4. A glaze of type "T," with both barium and zinc, is most favorable for opacity. This applies to tin oxide as well as the substitutes.

5. Where they can be used leukonin and antimony oxide are cheaper than tin oxide. Zirconium oxide is the best substitute for tin oxide if it can be procured at a sufficiently low price.

SOME DATA ON THE DEVELOPMENT OF TERRA COTTA GLAZES

By E. C. Hill

The study of Bristol glazes has received considerable attention in the past, and the extensive investigations made have covered the subject somewhat thoroughly. Bristol glaze mixtures are generally understood to be those containing feldspar, flint, clay, whiting and zinc oxide. The object of the work described herein was to study the effect of the addition to Bristol glaze mixtures of tin oxide, barium and magnesium carbonate, one or more of which are commonly used in the production of terra cotta glazes, and of strontium carbonate, not ordinarily used.

Part I

In the first part of the work, the additions were made to the eutectic mixture developed by Watts[1] in his study of Bristol glazes compounded on the eutectic basis. The molecular formula of this mixture is:

$$\left.\begin{matrix} 0.4\ KNaO \\ .3\ CaO \\ .3\ ZnO \end{matrix}\right\} \quad 0.60\ Al_2O_3 \quad 3.55\ SiO_2$$

and is the eutectic of three eutectic mixtures as follows:

KNaO	Al_2O_3	$6.4\ SiO_2$	feldspar and flint
ZnO	$0.318\ Al_2O_3$	$0.81\ SiO_2$	zinc oxide, clay and flint
CaO	$.348\ Al_2O_3$	$2.49\ SiO_2$	whiting, clay and flint

The glaze mixtures in this study were calculated from the molecular formulas. Canadian feldspar (molecular weight 557) was used. The clay mixture was composed of three parts English china clay and one part Kentucky ball clay (molecular weight for the clay taken as 258). The mixtures were weighed in 500-gram batches, ground with water for thirty minutes in laboratory jar mills, and sprayed on trials of a commercial terra cotta body

[1] *Trans. Am. Ceram. Soc.*, **18**, 631 (1916).

and engobe. Four trials of each were made, two being fired in a terra cotta kiln at cone 7 and two at cone $5^1/_2$. The cone 7 trials were fired in three different kilns but the cone $5^1/_2$ trials were fired together in one kiln.

In addition to the sprayed trials, portions of the glaze mixtures were dried, ground in a mortar and molded into test cones. The cones were fired in a gas-fired laboratory test kiln and the order of deformation observed closely. The cones were set on plaques in series and also with equal molecular equivalents of SnO_2, MgO, BaO and SrO. Four burns in all were made on the cones, and at least two burns were made on each mixture. Several cones of some of the mixtures were fired in each burn. The temperature of the kiln was rapidly increased to just below that required for the deformation of the cones, and as slowly as possible after the first mixtures began to deform.

Series A.—Tin Oxide

	A	A1	A2	A3	A4	A5
K_2O	0.40	0.40	0.40	0.40	0.40	0.40
CaO	.30	.30	.30	.30	.30	.30
ZnO	.30	.30	.30	.30	.30	.30
SnO_2	...	.05	.10	.15	.20	.25
Al_2O_3	.60	.60	.60	.60	.60	.60
SiO_2	3.55	3.55	3.55	3.55	3.55	3.55

The SnO_2 was not included in the RO of the formula. A is the eutectic glaze developed by Watts. In these burns it was a bright mat of creamish tint. The whiteness increases with the addition of SnO_2 to 0.10 equivalent (3.83 per cent tin oxide), but further additions increase the whiteness only slightly. The texture and sheen of A, A1 and A2 are about the same, but further additions of SnO_2, decrease the sheen of the mats. At cone $5^1/_2$, A5 was not well matured. All trials of A5 show small parting cracks, so that excessive amounts of SnO_2 may increase the crawling tendency of the glaze. However, more than 0.20 equivalent SnO_2 (7.40 per cent tin oxide in this series) is rarely used to increase the whiteness of glazes of this kind.

Cone Deformation.—A began to deform first and completed deformation at about the same time as standard cone 02, followed

by the balance of the series in order. Any addition of SnO_2 decreases the fusibility of A and the decrease is about in proportion to the amount of SnO_2 added.

Series B.—Magnesium Carbonate

	A2	B1	B2	B3	B4	B5	B6
K_2O	0.40	0.38	0.36	0.34	0.32	0.30	0.28
CaO	.30	.285	.27	.255	.24	.225	.21
ZnO	.30	.285	.27	.255	.24	.225	.21
SnO_2	.10	.10	.10	.10	.10	.10	.10
MgO	...	.05	.10	.15	.20	.25	.30
Al_2O_3	.60	.59	.579	.569	.558	.548	.538
SiO_2	3.55	3.47	3.39	3.32	3.24	3.16	3.09

Watts[1] determined the eutectic mixture of MgO, Al_2O_3 and SiO_2 to be $MgO{\cdot}0.392Al_2O_3{\cdot}2.00SiO_2$, which deforms at cone 12. The addition of MgO to glaze A2 (which is the same as Watts' eutectic glaze, but contains 0.10 equivalent SnO_2, not included in the RO) was made to conform to the eutectic mixture so that 0.392 Al_2O_3 and 2.00 SiO_2 were added for each equivalent of MgO. The addition was made by decreasing the molecular formula of A2 throughout by an amount equal to the addition of the equivalent MgO added, and adding the required amount of Al_2O_3 and SiO_2 to conform to the eutectic mixture. Thus, the formula of B1 (0.05 MgO) was obtained by multiplying the molecular equivalents of glaze A2 by the factor 0.95 and adding 0.05 MgO; $0.05 \times 0.392 = 0.0196$ Al_2O_3 and $0.05 \times 2.0 = 0.10$ SiO_2. The MgO was introduced as the "heavy" magnesium carbonate.

The texture of the series is that of a bright mat. The sheen increases somewhat with increase of MgO to 0.20 equivalent, but further additions decrease it. At cone $5\frac{1}{2}$, B5 and B6 are immature. B3 and B4 have some fine parting cracks and B5 and B6 have crawled badly. On this account, glazes of this kind should not contain more than 0.20 equivalent MgO and preferably not more than 0.10 equivalent.

Cone Deformation.—A2 deforms first, followed by the rest of the series in order. Any addition of MgO makes the glaze more refractory, and the increase is about proportional to the amount

[1] *Trans. Am. Ceram. Soc.*, **19**, 456 (1917).

of MgO added. Series B is more refractory than Series A. All cones of the A series are down before B3 (0.15 MgO) deforms.

Series C.—Barium Carbonate

	A2	C1	C2	C3	C4	C5	C6	C7	C8
K_2O	0.40	0.38	0.36	0.34	0.32	0.30	0.28	0.26	0.24
CaO	.30	.285	.27	.255	.24	.225	.21	.195	.18
ZnO	.30	.285	.27	.255	.24	.225	.21	.195	.18
SnO_2	.10	.10	.10	.10	.10	.10	.10	.10	.10
Al_2O_3	.60	.592	.583	.575	.566	.558	.549	.541	.532
SiO_2	3.55	3.57	3.60	3.62	3.64	3.66	3.69	3.71	3.73
BaO	...	.05	.10	.15	.20	.25	.30	.35	.40

Watts[1] found two mixtures of BaO, Al_2O_3 and SiO_2 that deformed at about the same time, but before any of the others. The one selected as a deformation eutectic, because of its rapid deformation, was $BaO{\cdot}0.43Al_2O_3{\cdot}4.01SiO_2$, which deformed at cone 6. The method of making the additions of BaO were the same as that used for MgO in series B.

Series C gave good white mats. The sheen decreases only slightly with increase of BaO.

Cone Deformation.—A2 deforms first, followed by the balance of the series in order. Any increase of BaO decreases the fusibility of A2, and the decrease is about proportional to the amount of BaO added. Series C is much more refractory than series A or B. All the cones of B series go down before C3 (0.15 equivalent BaO) deforms.

Series D.—Strontium Carbonate

	A2	D1	D2	D3	D4	D5	D6	D7	D8
K_2O	0.40	0.38	0.36	0.34	0.32	0.30	0.28	0.26	0.24
CaO	.30	.285	.27	.255	.24	.225	.21	.195	.18
ZnO	.30	.285	.27	.255	.24	.255	.21	.195	.18
SnO_2	.10	.10	.10	.10	.10	.10	.10	.10	.10
SrO	...	.05	.10	.15	.20	.25	.30	.35	.40
Al_2O_3	.60	.57	.54	.51	.48	.45	.42	.39	.36
SiO_2	3.55	3.37	3.20	3.02	2.84	2.66	2.49	2.31	2.13

Series D gave good white mats, the texture becoming more dull with increase of SrO. D7 and D8 are slightly immature at cone 7 and D5 to D8 are immature at cone $5^1/_2$.

[1] *Trans. Am. Ceram. Soc.*, **19**, 459.

Cone Deformation.—A2 deforms first, followed by the balance of series in order. Any increase of SrO decreased the fusibility of A2, and the decrease is about proportional to the amount of SrO added. This series is only slightly less fusible than the B series (MgO), but is considerably more fusible that the C series (BaO).

Series E and F.—Magnesium and Barium Carbonate

	A2	E1	E2	E3	E4	F1	F2	F3	F4	F5
K_2O	0.40	0.36	0.32	0.28	0.24	0.37	0.34	0.31	0.28	0.24
CaO	.30	.27	.24	.21	.18	.278	.255	.233	.21	.18
ZnO	.30	.27	.24	.21	.18	.278	.255	.233	.21	.18
SnO_2	.10	.10	.10	.10	.10	.10	.10	.10	.10	.10
MgO	...	.05	.10	.15	.20	.025	.05	.075	.10	.133
BaO	...	.05	.10	.15	.20	.05	.10	.15	.20	.267
Al_2O_3	.60	.581	.562	.543	.524	.586	.573	.559	.545	.531
SiO_2	3.55	3.50	3.44	3.39	3.33	3.53	3.52	3.50	3.49	3.47

In the series E and F, MgO and BaO together are added to A2. In the former, they are added in equal equivalents and in the latter, two equivalents BaO to one MgO. The results are about what would be expected from series B and C. All are good mats, except E4 and F5, which are immature at cone $5^1/_2$. E4 has some parting cracks.

Cone Deformation.—A2 deforms first, followed by E1. F1 is next, then E2. F5 is last to deform.

Series G and H.—Clay and Flint Variations

	F2	G1	G2	G3	G4	H1	H2	H3
K_2O	0.34	0.34	0.34	0.34	0.34	0.34	0.34	0.34
CaO	.255	.255	.255	.255	.255	.255	.255	.255
ZnO	.255	.255	.255	.255	.255	.255	.255	.255
SnO_2	.10	.10	.10	.10	.10	.10	.10	.10
MgO	.05	.05	.05	.05	.05	.05	.05	.05
BaO	.10	.10	.10	.10	.10	.10	.10	.10
Al_2O_3	.573	.523	.473	.423	.373	.523	.473	.423
SiO_2	3.52	3.42	3.32	3.22	3.12	3.22	2.92	2.62
Clay	0.233	0.183	0.133	0.083	0.033	0.183	0.133	0.083
Flint	1.01	1.01	1.01	1.01	1.01	.81	.61	.41

In series G, the clay is reduced by 0.05 equivalent in each member, starting with F2 (0.05 MgO·0.10BaO). In series H, the clay is reduced by 0.05 equivalent, in each member, starting with F2

and for each 0.05 equivalent of clay, 0.20 equivalent flint are taken from the flint content.

G1 is a good mat. The gloss increases proportionately with decrease of clay. G4 is a bright, glossy glaze. H1 is a good mat. The sheen becomes duller with decrease of clay and flint. H3 has a very dull texture, but is fully matured.

Cone Deformation.—G4 (0.033 equivalent clay) is the first to deform, followed in order by G3, G2 and G1. Decrease of clay increases the fusibility proportionately. H3 (0.083 clay, 0.41 flint) deforms first, followed by H2 and H1 in order. Decrease of clay increases the fusibility proportionately, even with decreasing flint. Comparing the two series, G4 and G3 deform before H3. H2 deforms at about the same time as G1. All deform before E2.

Series I and J.—Clay and Flint Variations

	F5	I1	I2	I3	I4	I5	J1	J2	J3	J4	J5
K_2O...	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24	0.24
CaO...	.18	.18	.18	.18	.18	.18	.18	.18	.18	.18	.18
ZnO...	.18	.18	.18	.18	.18	.18	.18	.18	.18	.18	.18
SnO_2...	.10	.10	.10	.10	.10	.10	.10	.10	.10	.10	.10
MgO...	.133	.133	.133	.133	.133	.133	.133	.133	.133	.133	.133
BaO...	.267	.267	.267	.267	.267	.267	.267	.267	.267	.267	.267
Al_2O_3..	.53	.48	.43	.38	.33	.28	.48	.43	.38	.33	.28
SiO_2...	3.47	3.37	3.27	3.17	3.07	2.97	3.17	2.97	2.77	2.57	2.37
Clay...	.29	.24	.19	.14	.09	.04	.24	.19	.14	.09	.04
Flint...	1.45	1.45	1.45	1.45	1.45	1.45	1.25	1.05	.85	.65	.45

As in series G and H, the clay and flint are reduced. The reductions are made in F5 (0.133MgO·0.267BaO). In series I, as in series G, any reduction of clay increases the gloss. I1 is a good mat, I4 and I5 are bright. In series J, as in series H, the reduction of clay and flint together decreases the sheen of the mats. J1 is a good mat, J4 and J5 are dull and have a curdled appearance.

Cone Deformation.—I5 deforms first, followed by I4, I3 and so forth. J5 deforms first in this series, followed by J4, J3, and so forth. The behavior of series I and J is similar to series G and H. From the results shown by E and F, we would expect

series G to be considerably more fusible than series I. Series G is more fusible on the high clay end of the series; but in the low clay glazes, series I is the more fusible. Thus when G_3 and G_4 were fired with I_4 and I_5, I_5 deformed first and I_4 and G_4 deformed close together. This may be due to the fact that the I series has a higher flint content.

Part II

The original outline of this study included only series A to J. The effect of the clay-flint variations led to the study of the effect on other RO, and to making the additions of SnO_2, MgO, BaO and SrO to a low-clay glaze instead of the one with 0.20 equivalent clay.

Series K and L.—Clay and Flint Variations

	A2	K1	K2	K3	A	L1	L2	L3
K_2O	0.40	0.40	0.40	0.40	0.40	0.40	0.40	0.40
CaO	.30	.30	.30	.30	.30	.30	.30	.30
ZnO	.30	.30	.30	.30	.30	.30	.30	.30
SnO_2	.10	.10	.10	.10	...	...	...	...
Al_2O_3	.60	.55	.50	.45	.60	.55	.50	.45
SiO_2	3.55	3.45	3.35	3.25	3.55	3.45	3.35	3.25
Clay	0.20	0.15	0.10	0.05	0.20	0.15	0.10	0.05
Flint	.75	.75	.75	.75	.75	.75	.75	.75

In series K, the gloss increases from A2, bright mat, to K_3, a bright, glossy glaze. In series L, the gloss increases from A, bright mat, to L_3, a bright glossy glaze. L_3 is transparent and shows the buff body of the trial. The opacity increases from L_3 to A.

Cone Deformation.—K_3 deforms first, followed in order by K_2, K_1 and A2. K_3 deforms with cone 03 and deforms at least one cone before A2. In the L series, L_3 deforms first at about cone 03, followed in order by L_2, L_1 and A. L_3 deforms at the same time as K_3, there being practically no difference in their deformation behavior. There is from one-half to one cone difference between L_3 and A. L_1 deforms before K_1 and A before A2, but there is practically no difference between L_3 and K_3. K_1, K_3, L_1 and L_3 were prepared again for a check. The clay in the original mixtures was three parts English ball and one part

Kentucky ball. In the check glazes, as well as those that follow, Florida kaolin was used as the source of clay. In the check series, L_3 deformed before L_1 and K_3 before K_1.

Series M and N

	M1	M2	N1	N2
K_2O	0.30	0.30	0.50	0.50
CaO	.35	.35	.25	.25
ZnO	.35	.35	.25	.25
Al_2O_3	.45	.35	.65	.55
SiO_2	2.85	2.65	4.05	3.85
Clay	0.15	0.05	0.15	0.05
Flint	.75	.75	.75	.75

Series M and N were made in connection with the check glazes K_1, K_3, L_1 and L_3. M1 is mat. M2 is transparent and glossy. N1 is a bright mat, N2 is transparent and glossy.

Cone Deformation.—Series M and N were fired with L_1 and L_3. Three burns were made in which these cones were fired on the same plaque and the order of deformation closely observed. In every case L_3 deformed first. L_1, M1, M2 and N2 deformed so nearly together that there is practically no difference in their order of deformation, and it is difficult to make any distinction between them. N1 deforms very slowly and is considerably behind the others. All of these mixtures, except N1, deformed before A.

Series O and P

	L3	O1	O2 = K3	O3	O4	P1	P2	P3	P4
K_2O	0.40	0.40	0.40	0.40	0.40	0.38	0.36	0.34	0.32
CaO	.30	.30	.30	.30	.30	.285	.27	.255	.24
ZnO	.30	.30	.30	.30	.30	.285	.27	.255	.24
SnO_2	...	.05	.10	.15	.20	.10	.10	.10	.10
MgO	...	...	...	...	...	.05	.10	.15	.20
Al_2O_3	.45	.45	.45	.45	.45	.45	.45	.45	.45
SiO_2	3.25	3.25	3.25	3.25	3.25	3.13	3.01	2.89	2.77

In series O, SnO_2 is added to mixture L_3. In series P, MgO is added to K_3, the same mixture as L_3 but with 0.10 equivalent SnO_2. "Light" magnesium carbonate was used. At cone 7, series O is glossy. At cone $5\frac{1}{2}$, it is a bright mat. There is little difference in the gloss or texture of these glazes, although

O4 has slightly less gloss than the others. Series P is bright at both cone $5\frac{1}{2}$ and 7. The entire surface of all the trials in this series are badly crawled or parted, which must be attributed to the use of the "light" magnesium carbonate, as none of the glazes in the other series crawled like these.

Cone Deformation.—L3, O1, O2 and O3 deform at practically the same time, with O4 only slightly behind. SnO_2 can be added to the low-clay glaze without decreasing the fusibility up to and including 0.15 equivalent SnO_2. In series P, any addition of MgO to K3 makes the mixture less fusible. All of the O series are deformed before P2.

Series Q and R

	K3	Q1	Q2	Q3	Q4	R1	R2	R3	R4
K_2O	0.40	0.38	0.36	0.34	0.32	0.38	0.36	0.34	0.32
CaO	.30	.285	.27	.255	.24	.285	.27	.255	.24
ZnO	.30	.285	.27	.255	.24	.285	.27	.255	.24
SnO_2	.10	.10	.10	.10	.10	.10	.10	.10	.10
BaO	...	.05	.10	.15	.20	...	...	...	...
SrO	...	...	...	...	...	.05	.10	.15	.20
Al_2O_3	.45	.45	.45	.45	.45	.45	.45	.45	.45
SiO_2	3.25	3.13	3.01	2.89	2.77	3.13	3.01	2.89	2.77

In series Q, BaO is added to K3. In series R, SrO is added to K3. Series Q are all good white mats, with the sheen decreasing slightly with increase of BaO. Series R is quite similar in color and texture to series Q, but the series has more sheen than Q, the sheen decreasing slightly with increase of SrO.

Cone Deformation.—In series Q, additions of BaO decrease the fusibility more than additions of either MgO or SrO. P1 and P2 are completely deformed before Q1 starts to deform. In series R, additions of SrO decrease the fusibility more than MgO, but less than BaO.

Summary

Bristol Glaze Mixtures.—The method of studying Bristol glaze mixtures used by Watts,[1] compounding the mixtures on the eutectic basis, is a convenient means of covering a considerable range of composition, and is no doubt more scientific than the usual method of studying glazes by direct variation of the molecu-

[1] *Trans. Am. Ceram. Soc.*, **18**, p. 631.

lar formula. However, in these complex mixtures, it is doubtful whether the eutectic method can be relied upon to produce the most fusible glaze.

The mixture developed by Watts is no doubt the best that can be produced from these mixtures as a bright, opaque, white glaze for stoneware;[1] it is not the most fusible mixture of the components. In series L of this study, it was found that this glaze could be made considerably more fusible by the reduction of clay. The low-clay glaze would not be satisfactory for stoneware, as it is transparent; but as a basis for introducing other materials used in the production of terra cotta glazes, it gave much better results than the eutectic glaze developed by Watts. Watts' study covered thoroughly the ground included between the eutectic mixtures used as the extremes of his investigation, and the data obtained are of great value. From the standpoint of the development of glazes for terra cotta there remains a considerable field of investigation open for the study of these mixtures. The area of fusible low-clay glazes was not touched in the eutectic investigation by Watts, since the mixtures with 0.10 equivalent of clay or less, contained 0.7 equivalent or more of feldspar. In the eutectic area the mixtures did not contain much more than 0.75 equivalent free SiO_2. It is quite likely that in the low-clay glazes, a further increase of SiO_2 would produce more fusible glazes.

Regarding the effect of clay on the Bristol glaze mixtures covered by series L, M and N in this study, it will be noted that in series M (RO = $0.30K_2O{\cdot}0.35CaO{\cdot}0.35ZnO$) there was practically no difference in the fusibility of the mixture whether the clay was 0.05 or 0.15 equivalent. In a study by Purdy,[2] with the RO = $0.20K_2O{\cdot}0.40CaO{\cdot}0.40ZnO$, mixtures with 0.16 and 0.20 equivalent were more fusible than any with less clay. In series L of this study, $0.40K_2O{\cdot}0.30CaO{\cdot}.30ZnO$, L3, 0.05 equivalent clay gave a more fusible mixture than any of the mixtures in the study. This mixture was somewhat more fusible than the same one with 0.15 equivalent clay, although the latter deformed as soon as any

[1] *Trans. Am. Ceram. Soc.* **19**, p. 301.

[2] *Ibid.*, **5**, p. 159.

in series M and N. In series N, $0.50K_2O \cdot 0.25CaO \cdot 0.25ZnO$, N2 0.05 equivalent clay, deformed at about the same time as the others, except L3, but N1, 0.15 equivalent clay, was more refractory than any in series M and L.

In these series, it is shown that as the feldspar is increased, additions of clay make the mixture less fusible, until in series N, 0.50 equivalent feldspar, there is a great difference in the fusibility when the mixtures contain 0.05 and 0.15 equivalent clay. In a study made by the writer[1] it was shown that in high-zinc Bristol glaze mixtures, any increase of clay decreased the fusibility of the mixtures considerably. It is evident then that clay increases the fusibility of the mixtures only when there is a considerable amount of CaO present, and in mixtures similar to those in this study, the CaO must be 0.40 equivalent or more. CaO is the only component of a Bristol glaze that reacts readily with clay to form fusible mixtures. The results of the L, M and N series point strongly to the conclusion that mixtures similar to L3, with equal amounts of CaO and ZnO and low clay, will be found to be more fusible than any high CaO high-clay mixtures possible with these components. A certain amount of CaO is essential to this type of glaze, however, since it cannot be replaced readily by any other components without decreasing the fusibility of the glaze.

Terra Cotta Glaze Mixtures.—The addition of SnO_2 did not affect the glaze to a great extent. Added to the high-clay glaze, it decreased the fusibility of the mixture somewhat, but additions up to 0.15 equivalent gave a good white color. The high-clay glaze with 0.25 equivalent SnO_2 showed some parting cracks.

MgO decreased the fusibility of both the high- and low-clay glazes. We would expect that MgO, in small amounts at least, would lower the deformation point as it is considered an active flux. Riddle[2] found it to be a much more active flux in a porcelain body than CaO; 0.20 equivalent MgO from "heavy" magnesium carbonate in the high-clay glaze showed parting cracks in the glaze surface.

[1] *Trans. Am. Ceram. Soc.*, **18**, p. 513.
[2] *J. Am. Ceram. Soc.*, 621 (1918).

Any addition of MgO from "light" magnesium carbonate caused crawling over the entire surface of the trial. The MgO series was more fusible than either the BaO or SrO series. MgO increased the gloss somewhat in the high-clay glazes and produced bright glazes throughout in the low-clay series.

SrO decreased the fusibility of the glazes in this study. The glazes had a good mat texture. It might be used to advantage to replace BaO wholly or in part in terra cotta glazes, since it gives a somewhat more fusible glaze, were it not for its high cost. The SrO series was more refractory than the MgO series.

BaO produced the least fusible glazes of the entire study. Any addition of BaO made the glaze considerably less fusible with each addition. The BaO series had a good mat surface, the surface growing duller with increase of BaO.

The order of deformation of glazes containing equal molecular equivalents of each was SnO_2, MgO, SrO, BaO in both the high- and low-clay glaze. None of these, even in small amounts, produced a more fusible mixture. Seger,[1] discussing the control of fusibility of glazes, states that the fusibility may be increased by making the RO more complex, and that the glaze can be made more fusible by replacing lime or magnesia with lime and magnesia. This rule did not hold true in this study. The smallest amounts of any of the additions did not increase the fusibility; the addition of SnO_2, MgO and BaO at the same time in small amounts decreases the fusibility. We know that the addition of ZnO to cone 4 mixture, $0.30K_2O{\cdot}0.70CaO{\cdot}0.50Al_2O_3{\cdot}4.00\ SiO_2$, increases the fusibility. It is likely that if a Bristol glaze mixture were overloaded with either CaO or ZnO, the addition of MgO, or possibly BaO or SrO, would increase the fusibility. But if the CaO and ZnO equivalent are present in the proportion to form a fusible glaze, it does not seem likely that the fusibility will be increased by the addition of any of the components used in this study.

The glazes in series O, P, Q and R are much more fusible than the corresponding glazes in series A, B, C and D. In the latter, the clay content is 0.20 to 0.25 equivalent; in the former, it is 0.05 equivalent. Since the addition of SnO_2, MgO, BaO and SrO

[1] Seger's "Collected Writings," Vol. II, p. 572.

to the low-clay glaze gave much more fusible mixtures than the same additions to the high-clay glaze we may state that neither SnO_2, MgO, BaO or SrO reacts readily with clay to form fusible mixtures. Combining this with the observations made regarding Bristol glaze mixtures, that neither feldspar nor zinc reacts readily with clay, we may state in mixtures containing all of these, in whatever amounts or combinations, the addition of clay will result in a less fusible glaze unless there is a considerable amount of CaO present. In this respect then, the behavior of CaO is entirely different from any RO elements ordinarily used in this type of glaze.

None of the trials of any of the mixtures in this study were crazed when drawn from the kiln. Crazing might develop in some of these mixtures on weathering the trials. None of the glazes developed a pink color,[1] which might occur under certain firing conditions, in glazes containing SnO_2 and CaO. None of the glazes were blistered.[2] Variations in the firing temperature, in the rate of firing, or in the kiln atmosphere, might result in the appearance of pink discoloration or blistering in some of these mixtures.

DEPARTMENT OF CERAMICS
RUTGERS COLLEGE
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[1] *Trans. Am. Ceram. Soc.*, **18**, p. 396.
[2] *Ibid.*, **18**, p. 398.

THE IMPORTANCE OF ANALYSIS IN PURCHASING RAW MATERIALS FOR THE GLASS INDUSTRY

By Alexander Silverman

The purpose of this paper is to bring to the attention of glass manufacturers the variation in composition of raw materials employed in the batch. It is presented to encourage constancy and thus permit, as nearly as possible, of the manufacture of a uniformly good product. Two raw materials may both be good but may differ in composition. Substitution of one for the other may spoil a glass unless proper correction is made for the difference in composition of the new material.

To obtain uniform results it is necessary not only to know the temperature of the furnace, duration of the melt, and so forth, but to keep informed as to the composition, form and size of raw materials and cullet. The technically trained man realizes these important facts, so the glass manufacturer should rely on his judgment in such matters. No new material should be substituted for one already giving satisfaction until the chemist decides that it can be introduced and that it will answer the purpose for which it is intended. In many cases a cheaper material can be utilized but unless the proper corrections are made in the batch the experiment may prove rather costly in the end.

The variations in composition cited in the following tables are not to be interpreted as a criticism of manufacturers of raw materials. Most of the materials are desirable for specific purposes, but there must not be a promiscuous substitution of one for the other.

The writer is indebted to the many manufacturers and dealers who were kind enough to submit analyses of their products. Letters were sent to all manufacturers known to the writer and the tables used in this article were, with but a few exceptions, compiled from data received.

Sand

Table 1, on composition of sands for glass-making, is condensed from Dralle.[1] Changes have been made in some of the limits so as to include American sands whose analyses were available.

[1] R. Dralle, *Die Glasfabrikation*, 1, 141–145 (1911).

TABLE 1.—GENERAL COMPOSITION OF GLASS SANDS

	The finer or so-called "quartz" sands Per cent	Plate glass sands Per cent	Window glass sands Per cent	Sands for chemical glassware Per cent
SiO_2...	99.57–99.915	98–99.70	91.50–99.30	87.10–99.9
Fe_2O_3..	0.0019–0.02	0.03–1.9	0.1–0.89	0.02–0.05
Mn_3O_4	0–0.02	0–0.015	0–0.064	.....
Al_2O_3..	0–0.17	0.04–2.13	0–4.90	0.02–7.41
CaO...	0–0.037	0–0.12	0–0.34	0–0.34
MgO..	0–0.24	0–0.10	0–0.40	0–0.60
Na_2O+ K_2O	0–0.042	0–0.17	0–0.10	0–3.49
Ignition loss.	0–0.103	0–0.32	0–0.92	0–3.74

. The next table (2) shows six sands[1] whose composition is so nearly uniform as to impel one to choose any one of the six.

TABLE 2.—CHEMICAL ANALYSIS OF HIGH-GRADE SANDS

	1 Per cent	2 Per cent	3 Per cent	4 Per cent	5 Per cent	6 Per cent
SiO_2.....	99.48	99.58	99.00	99.65	99.33	99.03
Al_2O_3.....	0.16	0.12	0.30	0.11	0.16	0.23
Fe_2O_3.....	.02	.02	.03	.02	.02	.02
CaO.....	.11	.13	.15	.12	.15	.21
MgO.....	.05	Trace	.00	Trace	.11	.05
Ignition loss.....	.13	.17	.21	.23	.20	.35

A glance at table 3 will, however, reveal a difference in the grain size of the six sands which is also an important factor.

It is well to consider both the chemical and the mechanical analysis. The form of the sand grains might be taken into account, so the magnifying glass also functions at times.

TABLE 3.—MECHANICAL ANALYSIS OF HIGH-GRADE GLASS SANDS

Sample number	>1.0 mm. Per cent	>0.5 mm. and <1.0 mm. Per cent	>0.2 mm. and <0.5 mm. Per cent	>0.1 mm. and <0.2 mm. Per cent	>0.01 mm. and <0.1 mm. Per cent	<0.01 mm. Per cent	Total sand grade[2] >0.1 mm. Per cent
1.....	0.5	21.6	75.6	1.3	0.3	0.7	99.0
2.....	0.0	6.1	88.4	5.1	.2	.2	99.6
3.....	.0	0.0	76.6	21.3	.3	1.8	97.9
4.....	.0	1.5	97.1	0.8	.2	0.4	99.4
5.....	1.6	11.2	85.7	.4	.1	1.0	98.9
6...A few grains		2.7	90.1	6.1	.5	0.6	98.9

[1] G. P. H. Boswell, "Notes on American High-Grade Glass Sands," *J. Soc. Glass Tech.*, 1, 147 (1917).

[2] Amount indicated in this column passed through a 16-mesh sieve and was retained by a 150-mesh sieve (Tyler standard).

The composition of sands, and other materials which follow, for specific purposes, has been discussed in the literature so it would seem unnecessary to repeat specifications here.

Soda Ash

By assembling the analyses of various brands of 58 per cent dense soda ash for sale in the United States the following limits (table 4) were obtained. To permit comparison an analysis of 48 per cent ash is also included.

TABLE 4.—COMPOSITION OF SODA ASH

	48 percent soda ash	58 percent soda ash
	Per cent	Per cent
Na_2CO_3	83.055	99.05–99.52
NaCl	11.992	0.24– 1.10
Na_2SO_4	4.698	.055–1.104
Fe_2O_3	0.016	0–0.02
Al_2O_3		0–0.002
$CaCO_3$	.191	0–0.10
$MgCO_3$	.032	0–0.06
SiO_2	.016	0–0.003
Insoluble matter	....	0–0.05

High sulphate and chloride content might prove objectionable for the manufacture of some glasses and not at all serious for others. The density of the ash is an important factor.

Pearl Ash

The next table (5) shows the composition of pearl ashes.

TABLE 5.—COMPOSITION OF PEARL ASH

	Stassfurt 96–98 per cent	Stassfurt 98–100 per cent	From plant ashes	Refined beet sugar potash
	Per cent	Per cent	Per cent	Per cent
K_2CO_3	97.15	99.45	40–75	90–95
Na_2CO_3	0.07	0.05	3–6	3–5
KCl	1.25	.06	1–10	1–3
K_2SO_4	...	.02	15–30	1–3
Water insoluble	0.06	...	2–4	...
H_2O	.31	0.40	4–10	...

The writer has communicated with various American manufacturers during the past year and submits the following analyses (table 6) which were received:

TABLE 6.—COMPOSITION OF AMERICAN PEARL ASH

	1 Per cent	2 Per cent
K_2CO_3	80.50	83.90
Na_2CO_3	0.36	...
KCl	2.54	4.20
K_2SO_4	0.09	2.60
Water insoluble	.01	0.25
H_2O	16.50	9.05

When this paper was read at the Chicago meeting of the American Ceramic Society last September, a number of members present stated that the analyses submitted (table 6) did not include the better grades of American pearl ash. The writer has since written to manufacturers mentioned in the discussion and ascertained that while they manufactured high-grade material during the war, it is not available at present (October, 1919).

What has been said regarding sulphates and chlorides under soda ash also applies to pearl ash.

Salt Cake

Variations in the composition of salt cake are evident from the following (table 7) limits, taken from nine analyses received:

TABLE 7.—COMPOSITION OF SALT CAKE

	Per cent
Na_2SO_4	89.12–98.00
NaCl	0.62– 5.25
H_2SO_4	.20– 1.66
Al_2O_3 0–0.27 / Fe_2O_3 Trace–0.21	Trace–1.25[1]
CaO	0–0.48
MgO	0–0.16
Insoluble residue	Trace–0.77
Water	0–3.00

The desirability of reporting Fe_2O_3 and Al_2O_3 separately should be brought to the attention of manufacturers, as the iron content, especially, should be known.

[1] Three manufacturers reported Fe_2O_3 and Al_2O_3 together.

Niter

Fifteen analyses were submitted by dealers in niter. The limits are shown in table 8. For comparison the composition of double-refined niter is also given.

TABLE 8.—COMPOSITION OF NITER ON AMERICAN MARKET

	Standard 15 samples	Double refined[2]
	Per cent	Per cent
$NaNO_3$	94.12–99.75	99.746–99.75
NaCl	0.02– 2.50	0.02
$MgCl_2$	.00– 0.289	.005
Na_2SO_4	.00– 1.06	.....
$Na_2B_4O_7$	.00– 0.147	.....
Moisture	.00– 3.32	0–0.22
Insoluble matter	.00– 0.35	0.003
B_2O_3	.006[1]	.006
CaO	.06[1]	.....
KNO_3	.00– 3.9	.....
$KClO_3$	.00– 0.69	.....
$NaIO_3$	.01[1]	.....
$CaSO_4$	.102[1]	.....
$MgSO_4$	.219	.....

[1] 1 analysis. [2] 2 analyses.

Limestones and Limes

Analyses of twenty high-calcium American limestones are shown in table 9.

TABLE 9.—HIGH-CALCIUM LIMESTONES

Twenty analyses

	Per cent
$CaCO_3$	96.15–99.40
$MgCO_3$	0.11– 4.00
Fe_2O_3	.12– 0.44
Al_2O_3	Trace– 0.52
$Fe_2O_3 + Al_2O_3$[1]	0.25– 0.84
SiO_2	.20– 2.12
SO_3	.00– 0.18

Limits for seven dolomitic limestones are shown in table 10.

[1] Reported together in five samples; in remaining analyses they were reported separately.

TABLE 10.—DOLOMITIC LIMESTONES
Seven analyses

	Per cent
$CaCO_3$	53.80–59.16
$MgCO_3$	39.50–45.47
Fe_2O_3[1]	0.38 and 0.23
Al_2O_3[1]	.20 and 0.28
$Fe_2O_3 + Al_2O_3$	.005– to 0.58
SiO_2	.10– to 0.69
Organic matter[1]	.21 and 0.94

[1] Reported in only two analyses.

Burnt Lime.—Table 11 contains the limits given in analyses of ten high-calcium burnt limestones.

TABLE 11.—BURNT LIME (HIGH-CALCIUM)
10 Samples

	Per cent
CaO	89.20–98.05
MgO	0.10– 2.00
$Fe_2O_3 + Al_2O_3$	.23– 0.94
SiO_2	.40– 2.30
SO_3[1]	.10 and 0.18

[1] Reported for two samples only.

The range for seven burnt dolomitic limestones is indicated in table 12.

TABLE 12.—BURNT LIME (HIGH-MAGNESIA)
7 Samples

	Per cent
CaO	55.62–60.02
MgO	39.50–41.67
$Fe_2O_3 + Al_2O_3$	0.16– 0.88
SiO_2	.05– 1.34
SO_3[1]	0.90

[1] Reported for one sample only.

Hydrated Lime.—For six high-calcium hydrated limes table 13 shows the limits.

TABLE 13.—HYDRATED LIME (HIGH-CALCIUM)
6 Samples

	Per cent
$Ca(OH)_2$	92.50–98.54
MgO	0.12– 3.50
$Fe_2O_3 + Al_2O_3$	.21– 1.00
SiO_2	.07– 2.00
SO_3[1]	.....

[1] Reported in one sample only.

In some samples only 60 per cent of the material passed through a 100-mesh sieve. In one sample 99.34 per cent passed through a 100-mesh sieve; 98.48 per cent through a 200-mesh sieve.

Four hydrated dolomitic limes varied according to table 14.

TABLE 14.—HYDRATED LIME (HIGH-MAGNESIA)
4 Samples

	Per cent
CaO	47.08–50.47
MgO	32.04–40.73
$Fe_2O_3 + Al_2O_3$	0.20– 0.51
SiO_2	.08– 1.17
Combined H_2O	16.52–17.44
CO_2	0.69– 1.96

Manganese Dioxide

The extreme variation in composition of manganese dioxide is shown in the limits given (table 15) for six samples.

TABLE 15.—MANGANESE DIOXIDE
6 Samples

	Per cent
MnO_2	70.00–86.25
MnO[1]	0.47
Fe_2O_3	0.25– 4.00
SiO_2	.52– 3.85
CuO[2]	0.01 and 0.03
NiO[1]	.30
Al_2O_3[1]	1.74
CaO[1]	1.73
MgO[1]	.20
BaO[1]	1.54
$K_2O + Na_2O$[1]	0.22
CO_2[1]	.63
S[1]	.23
P_2O_5[1]	.32
H_2O	1.85–13.33

[1] Reported for one sample only.
[2] Reported in two analyses.

Manganese dioxide may be purchased in any form from the finest powder to pea size. The color produced, or in other words, the decolorizing effect, depends on size and form as well as composition.

Talc

Four talc analyses showed variations within the following limits:

TABLE 16.—TALC
4 Samples

	Per cent
SiO_2	52.02–63.40
MgO	27.83–33.50
CaO	0.00– 6.01
$Fe_2O_3 + Al_2O_3$	.20– 2.18
H_2O	2.50– 4.44

A fifth analysis submitted showed no magnesia but 28 per cent Al_2O_3. Something would be likely to happen in a glass if an alumina-bearing mineral were substituted for a magnesia-bearing one simply as "talc" for "talc."

Feldspar

Feldspar variations are apt to be noticed quickly because of the use of this material in opal and alabaster glass manufacture. Eight samples from different localities varied as indicated in table 17. As alumina and potash are some of the factors which

TABLE 17.—COMPOSITION OF FELDSPAR
8 Samples

	Per cent
SiO_2	65.00–71.75
Al_2O_3	16.70–21.15
K_2O	8.19–10.60
Na_2O	1.10– 8.35
Fe_2O_3	Trace– 0.40
CaO	0.12– 0.56
MgO	.00– 0.15
Ignition loss	.05– 9.35

govern the color in white glasses one should never buy a new brand of feldspar without making the necessary corrections in the batch.

Conclusions

Analyses of many other materials might be added. Four samples of fluorspar vary from 93 to 98 per cent in CaF_2; also

in their SiO_2 and $CaCO_3$ content. It would seem from the evidence already furnished unnecessary to burden the reader with more facts.

Raw materials do vary in composition. One may be able to save money by substituting a cheaper material and doing so advisedly. One is likely to lose money by substituting it pound for pound when the composition of the new material varies considerably from that of the substance formerly employed.

In this age, where economy is such an important factor in industry, every saving in cost counts. When a new material is offered, ask for a sample and an analysis. Submit these to your chemist and have him advise you concerning the desirability of making a change. If you purchase the new material have the chemist check the composition from time to time.

Buy on analysis and see that corrections are made in your glass to keep it uniform. Uninterrupted operation and a uniformly good product should result.

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NOTES ON SHEET AND PLATE GLASS MANUFACTURE

By Edgar H. Bostock

The difficulty incident to contributing a paper to the Glass Division of the American Ceramic Society is that practically no technical work was done in glass in this country until very recently indeed; and of necessity all its developments have been commercial, or, more exactly, of the "get things done" method. As the growing needs of our country called for certain glass, we have filled the most obvious and profitable of these needs and have gone on importing glasses more difficult to make.

Even in those industries that we have established, a wealth of raw materials has lured us into wasteful methods and always into quantity and not quality production. It would seem that with the ideal fuel conditions that prevail with us, the best of sands and easy access to alkali productions, with the undoubted inventive genius among our engineers and adaptability of our labor, that we should now be able to make great progress in the production of better goods than we have heretofore produced. Particularly is this the opportune time as news is coming from Europe of the scarcity of labor and raw material over there, forcing up prices and making it possible for us to enter other lines of manufacture before this closed to us.

The writer's life and training has brought him into contact with the manufacture of flat glassware more particularly; and while I am not sure that this differs very much from other branches, still it is true that the chemist and physicist are almost unknown there. The progress made has been almost entirely the work of factory-trained men, and it is only within the last decade that the recently formed large combinations have made a start in the employment of search and research men.

The Sheet Window Glass Industry

The production of sheet window glass is very typical of the entire tale of American production and its future developments. Es-

tablished as a definite industry in the early 1800's according to the best practice of the times, for a century it was a wandering industry following cheap fuel, first the wood of New Jersey and the New York lake regions, then the bituminous coals of Pennsylvania and Pittsburgh, and, in the years succeeding the 80's, the natural gas of Ohio, Pennsylvania, West Virginia, latterly Kansas and Oklahoma. This rapid changing of location did not give time for developments of basic worth, and, with the exception of the adoption of the Belgian tank system of melting in 1888, no changes making for progress took place.

In 1894, John Lubbers, in New Kensington, Pennsylvania, was experimenting in the mechanical handling of cylinders and sheets in the flattening oven, and then conceived the idea of producing sheet glass mechanically. This process was based upon reproducing the methods employed by the hand blower mechanically. Many years and many millions of dollars were spent upon this process before it was commercially a success; but, as it eliminates the cost of highly skilled labor, it is now a great financial success and the process is used here and abroad.

The thought of all those who have studied the problem of sheet glass production always reverts to the drawing or producing of sheets directly from the molten mass without the intervention of any blowing or other process, and a great deal of work has been done and many patents granted and tried. Up to date the most successful would seem to be the Owens Company development of the Colburn process, which is producing glass in quantity and recently in fair quality.

The point to be overcome (other than the production of quality) is the elimination of "waves" in the glass due to it not being perfectly homogenous in the cylinder drawing processes, caused by the strain placed in glass when turned from cylindrical to sheet form, and in the Colburn process largely to the strain marks made when the sheet is turned from vertical to horizontal motion. Extrusion rather than drawing and direct production without turns would seem to be points of attack that promise results if persisted in.

The window glass factories seem to require very little help from the chemist, for with natural gas and excellent materials the writer

has seen glass melted in quality beyond compare. Perhaps they have not found or cared to use the ideal decolorants, and therefore, turn out glass of numerous green, yellowish and bluish tints due to slight impurities in the sands used.

There were, of course, certain specialties in sheet glass imported that recently we have had to produce, and thin, dry plate glass, microscopic slides and covers and similar thin goods are successfully made.

Fluted glass, so largely used in reflected lighting, is being made superior to the usual foreign article. This was flattened in Belgium in kilns heated with artificial gas, which would occasionally deposit a small film of "sulphur" upon the plastic sheet, invisible until the sheet was silvered when it detracted from the reflective value. Our glass flattened by natural gas, whose "sulphur" content seems nil, has a preferable surface.

One problem allied with sheet production so far seems to have escaped us—the making of colored sheet glass successfully. This is a complicated problem, for the chemist's help in furnishing formulas for the making of any and all colors does not entirely solve it. Due to the reheating in flattening a cylinder to a sheet, some queer problems in heat treatment are presented, especially in ruby glasses; and again when these colors are "flashed" or coated upon crystal glass, we have the problem of having the color coat and the body with about the same coefficient of expansion, so that both in working and in use they may stand temperature changes. Moreover, the manual difficulties under the conditions of producing sheets of even thickness are such that we have not yet solved them. At least two firms are bravely attacking these problems.

The Plate Glass Industry

The plate glass industry has been able to make a better showing of progress than most branches of the industry. Of necessity it is organized into large units with large companies controlling them, who have worked for progress. When selected and graded properly, their product leads the world. For glazing purposes it is superior, for melted from a little more refractory material, its

surface is harder and more resistant to abrasion by wind-blown particles or cleaning.

When the need arose our supply of optically perfect plate for instrument work proved ample.

Possibilities of Development

Possibly our greatest progress has been and needs to be the filling of our needs in the many small articles made from sheet glass by reworking. As instances we may mention what is known as non-shatterable or duplex glass for gas masks and eye-protecting goggles, wind shield glass for submarine chasers, airplane cowls, port holes, instrument faces and so forth. The most usual means of production is by welding two sheets of desired thickness with an included sheet of water white pyralin under pressure and steam heat. Why may not this included sheet be any desired color or carry the numerals of any desired dial and in goggles the necessary corrective color for the eyes?

There has been and must be more intensive development of machine beveling for thousands of beveled meter and instrument faces, for the trying of watch glass bevels and similar work.

Drilling for the insertion of posts, pivots and so forth is an important art. Recently the writer had occasion to investigate the large percentage of breakage in drilling flashed glass and was amazed to find how little information was in possession of those engaged in the work.

Production of light-correcting and diffusing glass from sheet glass by means of acid working and staining, whereby absolute control of color is obtained, should be developed.

One development of which the writer is aware promises an entirely new method of production of electric heat, by the use of a glass coated with a metal deposit which will carry a certain type current and heat the glass to any desired point.

The production of glasses for the filtering of different light rays is of great interest, and much work remains to be done in glass to allow only colors true to the spectrum to penetrate, and for the exclusion of ultraviolet and red, and also for X-ray work.

This paper would seem to indicate that we have done no work but that al together commercial; but curiously enough, to ou

credit is the only altogether new note in art glass, that known as opalescent and used in ornamental window work to reproduce absolutely any desired picture. For many years there had been glasses rolled which were mixtures of color with opal, but the late John La Farge doing window work in the 80's wondered if it were not possible to mix colors in glass while molten even as he would mix color on his palette. It was attempted and led to the establishment of a new industry which produced so numerous color and tone mixtures that anything in nature or art might be reproduced.

One of the greatest problems is the production of the proper types of light-directing and diffusing glasses. Prismatic glasses with various angles designed to turn the light into a plane parallel with the floor of any building have been worked out and largely used in practice. However, they are worked up and glazed frequently without reference to these rules, and the great defect to-day, other than this, seems to be that they are usually melted from common green-colored glass when at very little extra expense for decolorizing a clearer glass might have been produced, adding greatly to their efficiency.

Regarding diffusive sheet glasses almost the first study, and certainly the most exhaustive, was given to this during the development of the lighting plan for the Panama Pacific Exposition. All available glasses were studied and while the ideal glass was not made for the desired purposes a fund of knowledge was accumulated.

Two types of diffusive glass would seem to be called for, one with great specular reflection and one with little reflection but the maximum diffusive and transmissive values. A glass of the former type is available but the latter is still wanting.

It was found that the same thing is true of these glasses as was said of prismatic glasses; that no great care had been taken as to the color and luster of the glass. It would seem for the slight expense involved that manufacturers would try to produce a better quality glass in these lines.

Diffusion by means of deflection within the mass, as in varying type of light opal glass carrying deflective particles in suspension, has reached a high stage of development and for light-enclosing purposes works very well.

THE MALINITE PROCESS FOR THE PRODUCTION OF SILLIMANITE REFRACTORIES

By A. Malinovszky

The Theory of the Process

The Malinite process is a method of producing sillimanite refractories. The object of the process is to produce an extremely refractory material, having a neutral quality, for metallurgical structures, such as open-hearth and electric furnaces, converters, crucibles, and molds, where high temperatures and destructive slags are employed; also for the chemical industry and for chemical stonewares.

By this process any aluminous rocks or minerals can be converted into a stable compound of aluminum monosilicate, known in the ceramic art as sillimanite, and which resists high heat and slag. I have discovered that if aluminum silicate such as clays, halloysite, and other like aluminous rocks or minerals of wide occurrence are smelted in a cupola furnace (which I have specially designed for this purpose) together with a suitable reducing agent in proper amount, it is possible to regulate the volatilization of the silica by proportioning the amount of the reducing agent, so that it shall be sufficient to reduce all the silica, or to produce only a fractional reduction of the silica to any desired amounts, leaving only a part of the silica unreduced so as to convert all the charge into sillimanite, or only a part into sillimanite and the rest into fused quartz glass. It should be mentioned at this point that sillimanite or corundum does not combine with fused silica, nor with amorphous silica when it is available.

Preparing the materials for the fusing and charging the cupola are as follows: I take, for instance, the crushed raw material, and mix therewith a suitable reducing agent, as carbon, in the molecular proportion to the silica content in the rock or minerals as indicated in the following equations:

$$2(Al_2O_3 + 2SiO_2) + 6Fe_2O_3 + 39C = Al_4C_3 + 4Fe_3SiC + 32CO$$
$$Al_4C_3 + 4Fe_3SiC + O_{21} = 2Al_2O_3 + 4SiO_2 + 12Fe + 7CO$$

or

$$2(Al_2O_3 + 2SiO_2) + 3Fe_2O_3 + 24C = Al_4C_3 + 2Fe_3SiC + 2SiO_2 + 19CO$$
$$Al_4C_3 + 2SiO_2 + 2Fe_3SiC + O_{15} = 2Al_2SiO_5 + 2SiO_2 + 6Fe + 5CO$$

On the assumption that the above equations are all correct it is then evident that SiC is formed, and that the SiC gives up the carbon, though not as rapidly as if SiC were alone. It depends on the ratio of combined carbon and the effect of Si upon the quantity and condition of carbon. When SiC gives up carbon, leaving behind Si, this ignites spontaneously on coming in contact with the blast, forming first SiO and then SiO_2. The attraction between Si + O and then SiO + O is very strong, and much heat is produced by the combustion of silicon to silica.

The above equation of the combining reaction of Si and Fe is perhaps not the exact chemical combination that might take place, because we have for example ferrosilicon, which shows that Si is able to unite with Fe in all proportions. The iron may be absent and in that case more fuel must be added in the cupola. The proportions of the mixture may, however, be varied, as an excess of carbon over the calculated amount of silica is sometimes necessary in order to complete the reaction. The iron when added in the proportion from 3 to 6 per cent, as an ore or as an impure clay, can be mixed with the charge.

It should be made clear that the iron acts as a catalyzer, inasmuch as it accelerates the reaction and promotes more complete reduction of the silica. The iron alloys with the silicon, and it is thus that the silica is separted and volatilized, or sinks to the bottom of the furnace with the iron in the form of pure metallic iron or as ferrosilicon, or alloyed with copper.

When fusion commences in the first hot zone, or layer, reaction is taking place, and Fe, Si and C are set free. Consequently after this first reaction the rest of the reactions take place more rapidly. It is thus the iron which accumulates, as it is not eliminated from the charge by volatilization. It is very desirable that

titanium be present, as it detains the combination of iron with alumina, and, therefore, the iron can act continuously with the silica when the mass is brought to boiling.

It will be seen that the chemical process must go on continuously and consecutively as each thermic zone may permit it. This is evident because in two or three hours the temperature has reached the stage where the heaviest fumes of silica emerge and the heaviest deposit of SiO_2 takes place. This volatilized silica has an apparent specific gravity of 0.47 and is snow-white.

The volatilization of the silica depends upon the total amount of carbon and iron present, and also upon the temperature of formation and perhaps also upon the presence of other elements, to create conditions favorable to dissociating the SiO_2 into Si + O_2, and the formation of SiC, which later is broken up when in turn SiO_2 is again formed by reoxidation.

From my standpoint, as I conceive this chemical action, it must be evident that the formation of the carbides such as Fe_3C, SiC, Al_4C_3 and CaC_2 and others are gradual, and as quick as they are formed they are changed again to oxides. Only the Fe remains in a metallic state. However, some of it is expelled in the form of magnetite iron (Fe_3O_4) as fine particles carried out by the blast. The iron which is in a combined state with the material or fused mass is in a magnetic state and, therefore, renders the whole mass almost magnetic.

A very interesting test was made on this subject. We sent some of our material to be tested by magnetic separation to Dings Magnetic Separator Co., Milwaukee, Wis. They found the following results: Laboratory sample 270 (20 mesh).

	Per cent
"A" medium magnetic	0.73
"B" first high magnetic	88.50
"C" second high magnetic	6.37
"D" non magnetic	4.40

If all the results obtained by my experiments, observations, and my experience in my process are correct, then I freely can state that C or CO, solid or in gaseous state, does not entirely break up the SiO_2 into Si + O_2, but this is rapidly accomplished

in the presence of Fe, Mn, or Ti. The proper quantity of Si volatilization and carbide formation varies with the nature and amount of impurities contained in the rock or minerals, temperature, and hydroscopic state of the blast.

Operation of the Furnace

The charging of the furance is accomplished as follows: Figure 1 represents the furnace and blower. The outside shell of the furnace is of common iron such as an old boiler shell; or an old

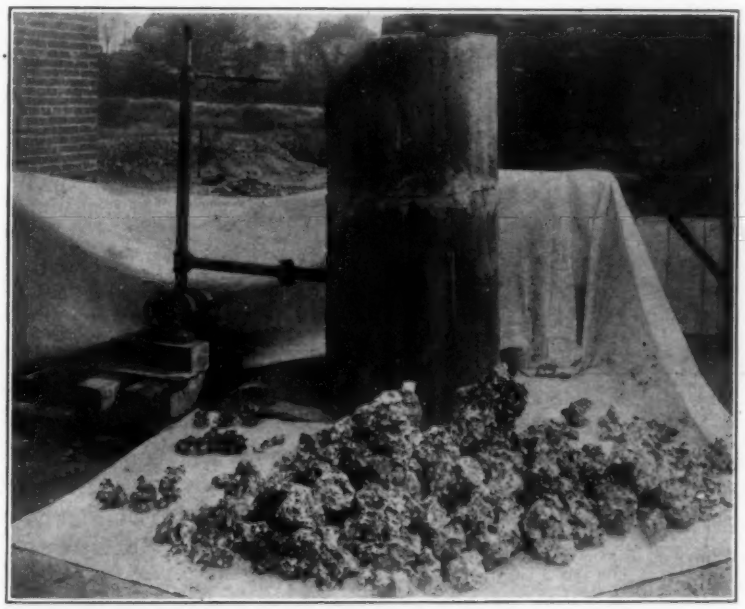

FIG 1.—Furnace with blower and fused product.

smokestack can be used. This is lined with good fire bricks. The bricks are then protected with a wall, about 4 inches thick, of the material which is to be fused, crushed fine. A mold is placed inside of the furnace, and the raw material is rammed all around between the inside of the furnace and the mold in a tapering shape, to about one inch thick on top. This will protect the fire bricks from becoming fused and at the same time will pre-

vent the fused charge from adhering to the fire bricks. Thus the fused mass can be discharged freely in a very short time. When the ramming is finished the mold is removed.

This protection of the raw material which is not mixed with coke is 4 inches thick only to the line where the mass shrinks to its limit. From this line it then tapers gradually to one inch at the top of the furnace. The reason for this arrangement is that when the furnace is fully charged and the heat commences to rise, the charge will shrink and sink down to this line, which is about four feet or so from the top of the furnace. The raw clay which is not mixed with coke or coal, and which I call a protective wall, will fall gradually on top of the charge and will hinder the free passage of the volatilized silica. To avoid this I usually mix the raw clay from that point on with a little coke. The top of the furnace from this line can be removed if so desired in order that the volatilized silica does not cling to the wall and become re-fused in the mass, which is a great detriment. The silica fumes should always pass off freely.

Large lumps of coke are placed on the grate bars for a height of about 30 cm. These big lumps of coke are used in order to protect the grate bars from fusing. Coke is one of the best solid fuels for my process as it does not crush or disintegrate and carries the weight of the charge better than coal. The coke serves also to preheat the air. On top of this coke about 60 cm. small coke is placed, then a little kindling wood, on top of this small coke again about 15 cm. high. Then the fire is lighted and the blower is also run very slowly until a heavy smoke commences to rise. Then the crushed clay mixed with the crushed coke is put into the furnace, and the speed of the blower is gradually increased to furnish about 18,000 cubic feet of compressed air per hour under three pounds pressure.

The steam commences to rise, mixed with heavy smoke, and in about two hours the charge commences to sink down and the gas to burst into flame above the cupola. The rest of the charge, about one-third, is then put into the cupola. The blast of compressed air is forced through the 165 cm. high fuel bed of coke, becomes very well preheated, and is then forced through the hot

mass, producing combustion. The silica commences to be deposited on the furnace wall. This period is very well characterized by the yellow flame coming from the furnace.

It is very important in my process to have the heat from the bottom of the furnace, and therefore, enough fuel should be placed

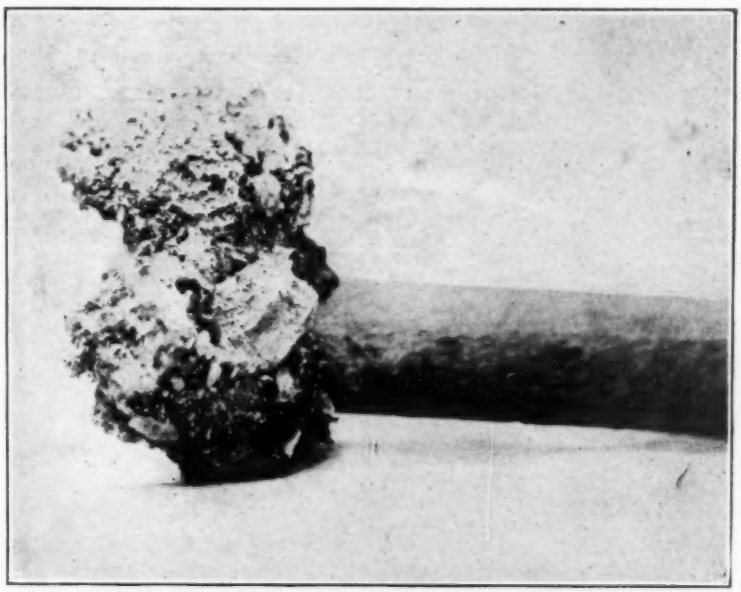

FIG. 2.—Rod, which was immersed in the fused boiling mass, covered with sillimanite crystals.

to last through the whole process. The furnace must be heated under such a reducing condition in the beginning as to convert gradually all the Fe, Si and Al into carbides. It should be understood that reduction takes place only at temperatures where solid carbon takes up oxygen from the material. The quantity of CO_2 present inside the furnace is sufficient to retard the process of reduction greatly, while the CO_2 is usually too small to be formed by the action of CO alone on the raw material. These facts point to the probability that there must be a considerable amount of reduction of the material taking place by the action

of solid carbon in the upper part or zone of the furnace and that the resulting CO performs an additional reduction in the still higher zone.

To promote or hasten the volatilization of silica from a material of high melting point such as halloysite, bauxite or any other aluminous rock or minerals, fluorspar or cryolite can used. I have found that cryolite is more desirable than fluorspar. Cryolite found in nature or the artificial variety can be used,

FIG. 3.—St. L. VI. B. taken in ordinary light. Note the development of sillimanite crystals and their variation in size from thin needles to comparatively large prisms. Note also the development and structure of the slag. Magnification—150 diameters.

or any other fluoride. If cryolite is used the volatilization of the silica is very rapid and the time is considerably shortened. Cryolite is a fluoride of aluminum and sodium $AlF_3 + 3NaF$. The action which takes place in the presence of cryolite with the silica is as follows:

$$2(AlF_3 + 3NaF) + 4SiO_2 = 3SiF_4 + Al_2SiO_5 + 3Na_2O$$

The liberation of the fluorine gas at a very low temperature from the cryolite produces the combination with silica, forming silicon tetrafluoride, which escapes as such in the gas form. During this reaction the raw material commences to break up and will be porous. At the same time the heat is increasing in the furnace,

FIG. 4.—St. L. VI. B. taken in plane-polarized light with crossed nicols. Compare with figure 3. Note the apparent isotropy of the thin sillimanite needles as against the strong birefringence of the thicker prisms.

liberating the carbon which will act more energetically to reduce the silica to silicon. The above chemical equation shows that by using cryolite Al is introduced and the product is increased by one molecule of sillimanite and one molecule less of the silica has to be driven off. By using cryolite a purer product is insured and this is accomplished in a shorter time. The quantity of cryolite used depends on the raw material and the purpose to which the product is to be put.

After three hours the whole charge is brought to a dazzling, glowing, white heat, the yellow-violet flames indicating the com-

bustion of Si to SiO and then to SiO_2, and the mass is then at its thermic action. The mass, or properly the content of alumina and silica, is boiling which fact is noticed by the roaring noise inside the furnace, and the silica is volatilizing in a dense white bluish, and lastly, in brownish fumes, for about two hours. At the end of five hours the whole process is completed, and air is

FIG. 5.—Ind. V. B. taken in ordinary light. Compare with figure 6. Note the size and development of sillimanite crystals imbedded in the interstitial glass and slag. Magnification—150 diameters.

blown into the cupola through the fused mass in order to produce cells resembling those of diatomacceous earth. The specific gravity of this fused mass varies from 2.62 to 2.86.

The furnace is provided with an exit pipe through which the fumes and gases are withdrawn and pass through into a settling system, tank, box, room or bags.

The hood can be removed freely to both sides and also lowered or raised. The hood is suspended above the furnace, and a

vacuum is created so as to collect all the fumes, and by this means the cupola is converted into a half vacuum furnace, or I may say blast on bottom and vacuum on top. The volatilized silica can be utilized for many useful applications in the arts. Its value is greater than that of the vitreous form.

By this process and system it should be clear that it is possible to effect a complete separation between the fused mass and alka-

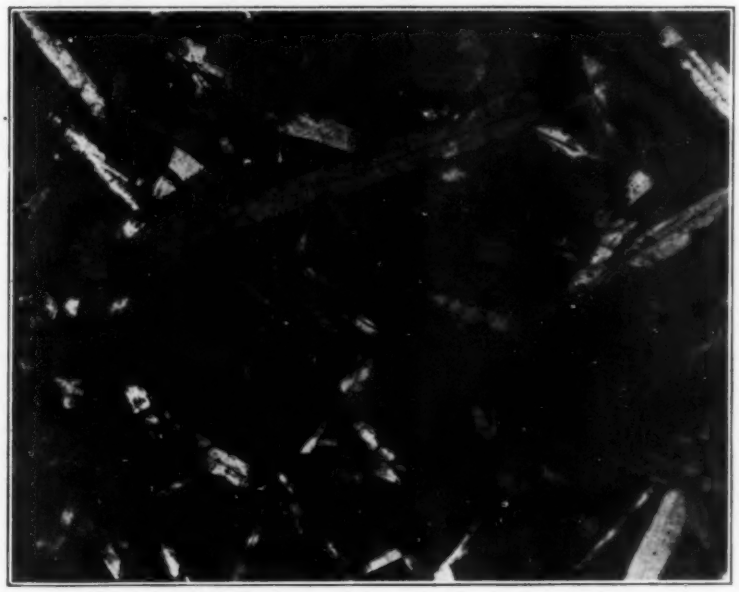

FIG. 6.—Ind. V. B. taken in plane-polarized light with crossed nicols. Note the isotropy of the glass and slag.

lies, and also silica if desired, in a more economical manner than by the use of an electric furnace.

The fused mass can be removed in different ways, the cupola being so designed as to accommodate varying conditions. The mass can be removed at the bottom with the cupola in place, or the cupola can be removed and leave the fused mass standing in place on the grate bars, or the mass and cupola can be removed, and another cupola, already charged, set up on the place, and

the operations can begin at once. With this system a continuous process is established. You will kindly note that the cupola can be taken apart without interfering with the process. Therefore, if batteries of cupolas are built, the fusion process is not hindered at any stage, and the whole process is more economical, as time is saved and the production is largely increased.

Properties of the Product

After the mass is removed it is broken up and ground to the required fineness, the iron having been separated out by a magnetic device. It is very interesting to note that almost all the iron and copper collects on the bottom of the fused mass in big nodules. From the top to the center, the mass is almost entirely free from iron and copper. The iron on analysis showed that it is alloyed with silicon, copper and zinc; a small amount of gold was also found.

The fused mass obtained by the process is a product of a very high refractory quality, free from expansion and contraction at any degree of temperature, and it will not warp, crack, nor spall from changing temperature.

Sillimanite so produced by the separation from such a highly viscous magma solution at such high temperature without the addition of any flux is a stable compound of alumina and silica. Research in thermal investigations by many investigators has shown the existence of only one aluminum silicate formed from fused mixtures in crystal form. This is sillimanite, Al_2SiO_5, with a melting point of 1815° C.

It is not necessary to state that fusion tests made on the product of this cupola at different places showed many times that Orton cone 39, and in one case even cone 40, was fused and the cone made of the Malinite product remained unaffected. Melting points of cones 36, 37 and 38 are common from the fused mass.

Three sample bricks were placed in a high-temperature electric furnace, in contact with the carbon resistor plates. The electric current passed from plate to plate and there was often an arcing action of certain points, which would cause a local temperature of 6300° F. The furnace temperature was approximately 3000° F., and under these conditions the Malinite brick was unaffected.

Our experience with magnesite bricks in this furnace has been that they scale off and disintegrate under the furnace conditions explained above.

Investigations made by different petrologists have demonstrated from different fused masses of different fusions that the crystals present have indentically the same optical properties as the crystals of sillimanite found in porcelain bodies. The

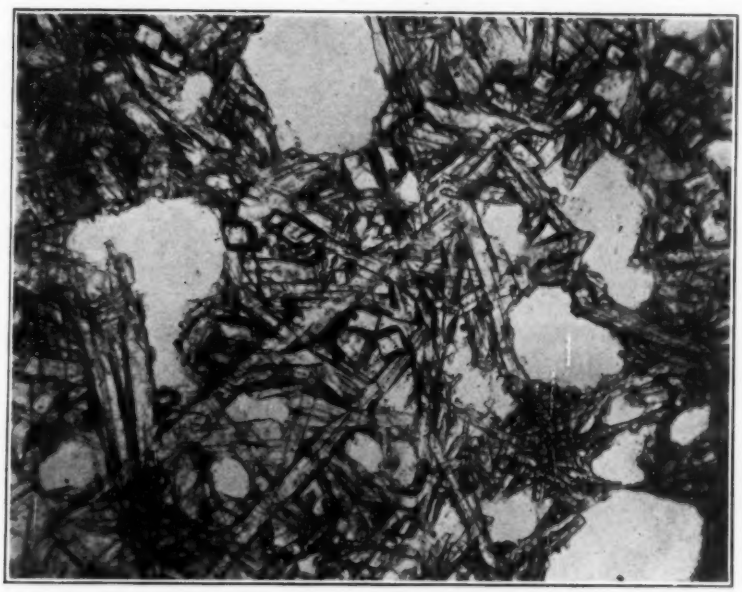

FIG. 7.—St. L. C. II. No. 13 (1) taken in ordinary light. Note the sillimanite coarse crystal development, the int rstitial glass and the almost complete absence of slag. Magnification—150 diameters.

cuts demonstrate the fibrous form of the coarse crystals, aggregates of long unusually strong acicular crystals with parallel extinction and positive elongation. The interference colors are bright.

I have found in this work that the rate at which this crystallization proceeds depends a good deal on the temperature and fluidity of the magma solution from which these crystals develop. It seems to me that the created temperature of the thermal action

of alumina and silica increases the fluidity of the mass to such an extent that a molecular concentration of alumina and silica takes place, and on cooling from this high temperature the sillimanite separates from the mass. When enough silica is driven off by volatilization and only one molecule of silica is left behind in the mass in proportion to one molecule of alumina, sillimanite will separate out and the mass is converted wholly into sillimanite. If more of the silica remains in the mass, then sillimanite and glass are formed. This glass, however, may be the same composition as sillimanite, but even if it should be fused quartz, it is not a detriment to my product, because sillimanite remains uncombined and will always separate out as such.

Should the magma be siliceous and contain calcium, sillimanite formation is retarded or diminished. In this line I did some very interesting work. I took precipitated alumina, silica and calcium and mixed them in the following proportion:

	A	B	C	D	E	F
SiO_2.....	36.1	35.3	34.5	33.6	11.1	32.2
Al_2O_3.....	61.5	60.0	58.6	57.3	56.0	54.8
CaO.....	2.4	4.7	6.9	9.1	32.9	13.0

These mixtures were made into small briquets and burned to cone 14, some were burned twice. After they were cool, small pieces were broken off and examinations made under the microscope. These crystals showed different optical properties than was the case in the former fusions; the refractions were not so strong.

When treated with hydrofluoric acid the samples marked D, E, F were dissolved and a residue of powder left, which on analysis were found to be alumina, calcium and a trace of silica. The silica was almost all volatilized. This demonstrated that the crystals noticed under the microscope were not sillimanite (Al_2SiO_5) but that they were wollastonite ($CaSiO_3$).

The pieces which did not contain lime were not attacked at all by the hydrofluoric acid treatment. The pieces marked from A to C were a little attacked by the acid.

I also made a very careful study and investigation of the formation of sillimanite with pure kaolin, fire clays and halloysite, but have failed to find sillimanite so far in the above samples

burned in a commercial kiln. Numerous statements are given and published, in text-books and other literature, that clay decomposes into Al_2O_3 and SiO_2. LeChatelier, Mellor and Holdcroft, Sosman, Klein and other investigators state that china clay decomposes and gives a product of free silica, alumina and water. The former two, however, are said to recombine in part again to

FIG. 8.—St. L. C. II. No. 12 (1) taken in plane-polarized light with crossed nicols. Compare with figure 7. This photograph accentuates the difference between the sillimanite crystals and the isotropic glass.

form sillimanite when heated above 1200° C. I have tried with two different washed kaolins, four different fire clays, and three different halloysites to produce sillimanite. The kaolins were purchased from Roessler, Hasslacher Chemical Company, the halloysites came from Indiana, Georgia and Alabama, and the fire clays from Olive Hill, Mount Savage, Ohio and St. Louis, Mo.

These materials were ground and made up in small briquets of $^1/_4$ inch size, dried and heated to Orton cone 14 for the length

of 96 hours. After they were allowed to cool, chips were broken off for microscopic examination. No sillimanite was noticed, except in very small pockets in the St. Louis fire clay. A few very fine crystals were visible with a powerful lens, but they were very uncertain to be sillimanite.

The samples were then placed in the kiln again, on the back wall, and heated four more times to cone 16, and one time to 18. After they were allowed to cool to atmospheric temperature, small chips were broken off again and microscopic examinations made. No signs of sillimanite crystals were found in the kaolin, nor in the halloysites, nor in the Olive Hill and Mount Savage clay, but the St. Louis fire clay and also the Ohio fire clay contained a considerable amount of fine crystals in nests scattered throughout the body.

This demonstrates that fire bricks made of pure fire clay, free from alkalies, have to be heated above 1400° C. in a prolonged heat and then allowed to cool slowly to form a few crystals of sillimanite. The formation of the sillimanite crystals in fire bricks is produced only by prolonged heating at high temperature, and is remarkably better developed in the presence of feldspar minerals and micas in the clay.

Mr. A. A. Klein made a very careful investigation in this line on washed North Carolina kaolin and on English kaolin and found small crystals of sillimanite in the two clays heated in a platinum resistance furnace for five hours between 1400 and 1450° C.[1]

After I had mixed the raw kaolin, halloysite, and fire clays with 25 per cent feldspar and subjected the mixtures to high temperature, cone 14 for 96 hours, they were allowed to cool and chips were broken off. It was found by microscopic examination that in every sample crystals were noticeable in abundance.

The circumstantial evidence of this work demonstrates that the work done by Vernadsky, Zoellner, Klein and others prove my statements, that in a porcelain body the chief cause of the presence and the separations of the sillimanite crystals from the magma solution, when allowed to cool down slowly from the high temperature of 1400° C. to ordinary atmospheric temperature,

[1] Bureau of Standards *Tech. Paper* 80.

is the solution of the kaolinite by feldspar and more so by orthoclase than by albite. Therefore, in this case kaolin and feldspar are the only essential materials to create conditions for the production of sillimanite crystals.

Vernadsky found that sillimanite crystals are produced whenever mixtures rich in kaolin and feldspar are subjected to a tem-

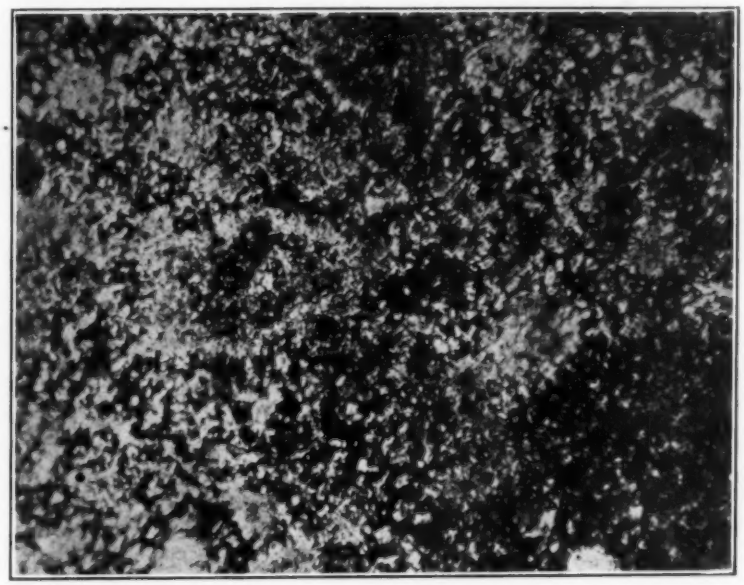

FIG. 9.—St. L. C. II. No. 13 (2) taken in ordinary light. Compare with figure 10. Note the small mainly anhedral sillimanite crystals, the opaque slag and the glass sometimes rendered "muddy" by amorphous sillimanite inclusions. Magnification—150 diameters.

perature over 1300° C. The velocity of crystallization of sillimanite in alkaline bodies is governed by time, temperature, and the rate of cooling. In porcelain bodies the slower the cooling the more complete is the process of crystallization, as by this slow-cooling process the corresponding chemical composition arranges itself into crystals; a part of the liquid will recrystallize before the remaining magma commences to solidify. In quick cooling of a

porcelain body or of fire brick the magma solidifies so fast that the chemical components have not the time to form crystals.

In the late work of Albert B. Peck, published in the JOURNAL OF THE AMERICAN CERAMIC SOCIETY, **2**, 175 (1919), he also states, as do Plenske and Zoellner, that the size of the crystals developed appears to be influenced by the three factors; the amount of feldspar or other fluxes present; the mobility of the feldspar, as affected by the temperature of firing; and the length of time of firing. By my process the crystals are developed in a few hours and cooled in about one hour, and unusually large and coarse crystals of sillimanite not known to the present are produced.

Samples subjected to the hydrofluoric acid test have given the following results:

Sample	Length of treatment	Per cent loss
19	1 day	180
20	3 days	170
21	6 days	18.5
22	6 days	4.0
23	12 days	5.96

Samples 22 and 23 were chips from the fused product as it came out from the furnace. The average analysis of these two samples was as follows:

$$SiO_2 = 37.1 \text{ per cent}, \quad Al_2O_3 = 62.9 \text{ per cent}$$

These crystals once formed are stable and cannot be destroyed in commercial furnaces. But we know that in fire bricks, the bricks become gradually weaker mechanically, as the crystals are formed and the alkalies volatilize. I have noticed in fire bricks made from fire clay, that when they are exposed to repeated heating to high temperatures, and allowed to cool slowly, the small amount of lime which the clay contains does not hinder the formation of sillimanite. But as soon as sillimanite commences to be formed, alkalies and some of the silica commence also to volatilize. The volatilization of silica is especially noticeable in the presence of solid carbon when the furnace is under reducing condition. This applies to all refractories containing silica, even bauxite or flint.

Therefore, it can be said that all the refractories made from fire clay, flint clay, or bauxite are not stable compounds for linings in furnaces, as they are exposed to continuous expansion and contraction and chemical action. Therefore, their life is limited at prolonged high temperatures at which they suffer mechanical

FIG. 10.—St. L. C. II. No. 13 (2) taken in plane-polarized light with crossed nicols.

destruction. Under the prolonged high temperature, the feldspar mica and lime present in the clay commence to fuse at 1200° C., as demonstrated by Le Chatelier and Bogitch, forming solutions which cause softening and so dissolve the more refractory substances, leaving cavities for accumulation of ashes, carbon and other substances. This causes further destruction of the brick until they gradually melt away. The vapor of different substances and the volatilized alkalies from the fuel and materials also, cause fluxing actions on the surface of the brick, forming solutions from which the sillimanite is more apt to separate.

The fused part goes continually deeper and deeper into the brick.

All this crystallization process occurring in the bricks during their use is very irregular and is always accompanied by contraction in volume. At the same time local strains are set up through the whole brick, causing rupture of the brick, or may be the whole wall has to give away. The experimental evidence of the many

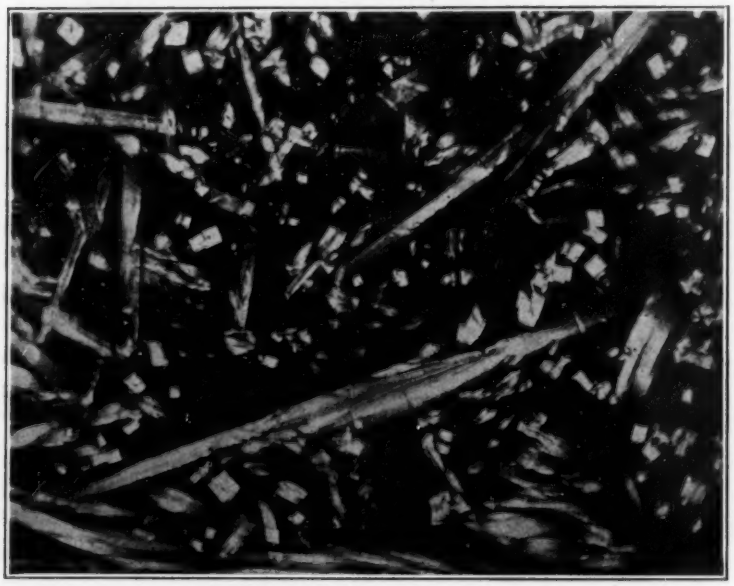

FIG. 11.—St. L. C. III. No. 14 taken in ordinary light. Compare with figure 12. Note the presence of predominating sillimanite and the size as well as the development of the crystals of this constituent. Note also the practical opacity of the glass rendered so by the comparative thickness of the section. Magnification—115 diameters.

tests made at different places on Malinite is proof of the stability of the sillimanite crystals produced by my process.

It is of capital importance in the manufacture of refractories to have always a pure uniform material. We know this is very difficult to do with the present methods of working clay; perhaps one strata of the fire clay is very pure while the adjoining

strata is contaminated with materials which make the clay worthless for refractory purpose. It can be seen that with my process it is very simple to overcome this trouble, and a uniform material is always obtained. Plans can be better and more systematically arranged with a perfect control of the raw material at every point and stage in the manufacturing operations. The variation in raw material from time to time is avoided and the process at every stage is brought under full control.

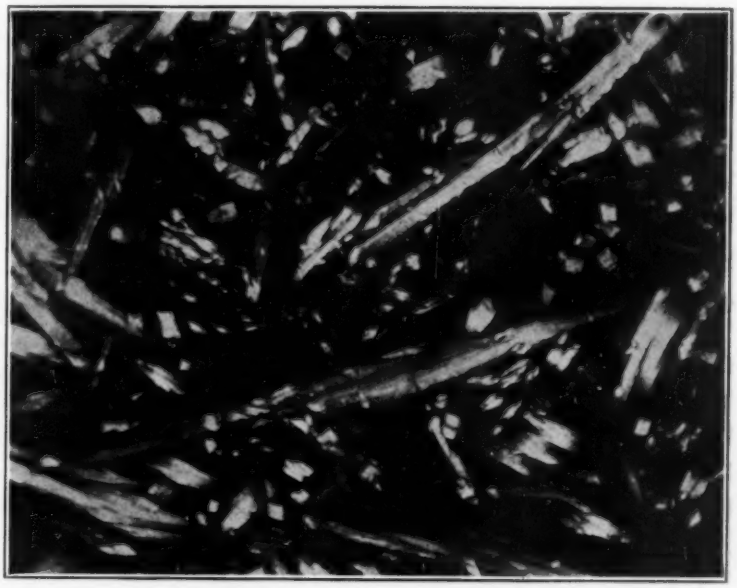

FIG. 12.—St. L. C. III. No. 14 taken in plane-polarized light with crossed nicols.

Here I want to illustrate the analysis of raw and cupola product after the raw materials are fused. The first analysis is from halloysite. You will kindly notice the higher per cent of lime and magnesia, also the presence of some titanium. I am very certain that no titanium is present in this halloysite in the raw state, as I made numerous analyses, I may say thirty or more, and never found titanium. I found cobalt, zirconium and manganese in

traces, but never titanium. The titanium in this analysis is brought in by the impurities of the coke. We used a coke which showed on analysis 15.50 per cent of ash, called Turkey Knob coke.

	Raw halloysite	Calculated. The moisture is omitted	The fused product
SiO_2	34.06	47.54	17.16
Al_2O_3	33.07	46.16	78.39
Fe_2O_3	3.11	4.35	.89
CaO	.49	.68	1.50
MgO	.42	.59	.29
Alk	.49	.68	0.36
TiO_2	.....	.....	.....
Moisture	28.53	.....	.....
	100.17		

St. Louis fire clays which we have received:

	1	2	3
SiO_2	47.25	52.20	53.50
Al_2O_3	28.22	26.92	28.75
Fe_2O_3	4.13	2.68	3.84
CaO	.70	1.00	1.60
MgO	1.77	1.88	0.87
Alk	.76	0.51	...
TiO_2	4.55	4.10	4.10
H_2O	3.80	10.70	7.30
Volatile matter	8.82		

In the third analysis the alkaline content is missing, as it was not sought for.

The analysis of the fused product from St. Louis fire clays are as follows:

SiO_2	32.60	33.10	38.10	36.30	37.50
Al_2O_3	63.84	63.59	56.17	58.95	57.94
Fe_2O_3	1.62	1.57	2.61	2.40	2.16
CaO	.89	.63	1.10	.65	.80
MgO	.32	.21	.49	.22	.22
TiO_2	.67	.82	1.12	.95	1.10

The specific gravity of the fused mass varies from 2.04 to 2.20 and in some cases to 2.66. The brick, $9 \times 4^1/_2 \times 2^1/_2$ inches, weighs 4.280 grams or 9.416 pounds and have an apparent

gravity of from 2.40 to 2.69. The analysis of the volatilized silica is as follows: SiO_2 94.5, Al_2O_3 0.50, CaO 0.60, MgO 0.51, alkalies 3.67, and the specific gravity varies from 0.471 to 1.875.

The fused products when removed from the furnace were carefully weighed after every fusion. The charge of raw material placed in the furnace weighed 600 kilograms. The average product taken from the furnace after the fusion was completed weighed 328 kilograms. This contained an average of 40.85 kilograms of iron, which had an average analysis as follows: Si 3.80, Al 1.20, Fe 44.86, Ti 49.14, Cu 0.09, and C 0.89. Many big pieces resembling copper coated with titanium, with a red copper metallic luster, were found. In metallurgy practice it is well known that titanium cyanonitride, Ti_5CN_4 is formed in the slag of blast furnaces in which titaniferous iron ore is worked, and that it is found in the form of cubes with copper-red metallic luster.

Microscopic Examination of Samples

The following is a report made by Mr. A. A. Klein on the microscopic examination of some of the products made by this process.

St. L. VI. B (see figures 3 and 4).—This sample was a gray sintered one showing a great number of fine spheroidal pores. The constituents found were sillimanite, glass, slag, and an extremely small amount of amorphous sillimanite.

A study of the microstructure by means of a thin section showed that there were two distinct types depending apparently upon the states of sub-division of the raw materials entering into the product. Such a condition is also found in other types of refractories. That portion of the product formed from coarser raw material contained, in general, fine sillimanite in intergrown fibrous crystals which sometimes were too thin to show double refraction. Amorphous sillimanite was also found here.

The main portion of the sample, however, consisted mainly of crystallized sillimanite occurring in the characteristic long, prismatic crystals which varied in length from under 0.01 mm. to 0.5 mm. These were embedded in a low refracting, colorless to yellow glass and in a black opaque slag.

The sillimanite needles which occurred distinctly intergrown and intertwined were longer and thicker than those normally found in porcelain bodies, but their optical constants were identical with those given for artificial sillimanite, namely, α = 1.638, β = 1.642, and γ = 1.653. The character of the principal zone was positive. The extinction was parallel and the inter-

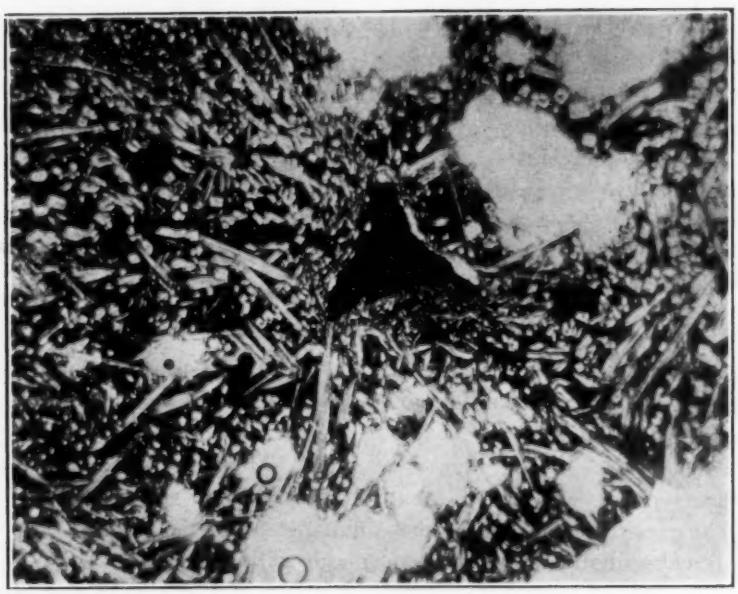

FIG. 13.—St. L. C. III. No. 14 taken in ordinary light. Note the opaque material at the center most of which possesses a metallic luster. This photograph gives a bird's-eye view of the production at low magnification—25 diameters.

ference figures were biaxial with a positive optical character and a small optic axial angle. There was nothing found optically that would indicate another modification of sillimanite, nor even the presence of any great amount of extraneous oxides held in solution.

A quantitative determination of the index of refraction for the glass gave a value for sodium light equal to 1.510 ± 0.003. The

opaque slag occurred generally in irregular forms which showed no metallic luster but rather appeared dull in reflected light.

Ind. V. B (see figures 5 and 6).—Visually, as well as microscopically, this sample rather resembled the product previously described. The constituents were sillimanite, glass, slag, and amorphous sillimanite.

As in the case of the previous sample, the microstructure was of two types: first, a uniformly fine crystalline one which was formed from original coarse particles of raw material, and, secondly, the predominant, distinctly coarser crystalline type which was formed from the reaction of materials in a comparatively fine state of sub-division.

The former type generally consisted of very small sillimanite needles which rarely exceeded 0.05 mm. in length and were generally associated with relatively small amounts of amorphous sillimanite. In a few instances, however, the amorphous sillimanite was the chief constituent.

As stated above, the coarser crystalline type was predominant and here the chief constituent was crystallized sillimanite which occurred in a ground mass of colorless glass and opaque slag.

The sillimanite occurred mainly in well-developed crystals of length varying from about 0.01 mm. to 0.08 mm. Optically the crystals were identical with artificial sillimanite. The glass was of a slightly lower index than in the previous sample, the value for sodium light being 1.503 ± 0.003.

St. L. C. II (see figures 7, 8, 9 and 10).—There were two distinct products found in this sample: (1) A white to grayish white vitreous body; (2) a grayish brown sintered product which occurred to a greater extent than (1).

(1) The constituents here were sillimanite with smaller amount of colorless glass and a very small amount of opaque slag.

The sillimanite crystals were well developed and occurred as much intergrown prisms which attained a maximum length of about 1 mm. Optically, this material agreed with the constants found for artificial sillimanite. In the interstices between the sillimanite crystals and acting as a ground mass for them, oc-

curred the colorless glass which was present to an extent of at least 30 per cent. The refractive index of the glass for sodium light was 1.514 ± 0.003.

(2) The constituents here were sillimanite, amorphous sillimanite, alpha alumina, slag and glass. The sillimanite crystals were generally anhedral, irregular and showed prismatic development to only a slight extent. The largest crystal measured

FIG. 14.—I. Ind. taken in ordinary light. Note the extremely coarse crystals of sillimanite and the globules of opaque material. Magnification—25 diameters.

under 0.2 mm. in length and most of them were under 0.02 mm. Despite their minuteness and due to their relative thickness, practically all showed double refraction. The refractive indices, which were the only constants that could be obtained with high accuracy, agreed with those given for artificial sillimanite.

About 15 per cent of alumina was found in small anhedral crystallites intimately associated with the fine crystalline aggregates of sillimanite. Some of these attained 0.1 mm. and in

all the determinable optical constants agreed with those found for alpha alumina. As in the products previously described, the colorless glass and the slag associated with it occurred as a matrix cementing the crystals of sillimanite and corundum.

St. L. C. III (see figures 11, 12, and 13).—This sample was a grayish black slag-like product with a stony appearance. It was quite porous, the voids being irregular to spheroidal and

FIG. 15.—I. Ind. taken in plane-polarized light with crossed nicols. Compare with figure 14. Note the two sets of parallel grouped sillimanite crystals one of which is practically in complete extinction.

filled in some instances with thin white to brassy yellow globules possessing a metallic luster. Their chemical composition is unknown but they appear to be at least in part ferrosilicon.

The constituents of the grayish black stony product were sillimanite, glass and the opaque material with metallic luster.

The sillimanite occurred as anhedral long, colorless, prismatic crystals which optically agreed with the data found for artificial

sillimanite. The crystals varied in size and reached a maximum length of 1.7 mm.

The glassy matrix was colorless to greenish and of a refractive index for sodium light equal to 1.510 ± 0.003. The slag occurred intimately associated with the glass in the form of minute dusty particles.

The opaque metallic constituent was found in rounded to irregular masses. With reflected light a portion of these at least were found to have metallic luster although in some cases the major portion of the grain was dull. It is impossible to state from this examination just what the actual chemical composition is, except that the material is in part metallic and may, therefore, contain such elements as iron, copper, silicon and so forth.

I. Ind. (see figures 14 and 15).—This was a grayish white, fused, vitreous, fibrous product. The constituents were sillimanite, glass and opaque material. The sillimanite was in the form of long, fibrous crystals whose length was limited only by the size of the sections, namely, 8 mm. These fibers showed a tendency toward parallel grouping and in a few instances an angle of approximately 15° was included between two sets of grouped fibers.

The glass occurred as the matrix between the sillimanite crystals as well as in thin stringer-like inclusions parallel to the long direction of the sillimanite fibers. The refractive index was approximately the same as in some of the products previously described: namely, 1.515 ± 0.003.

The opaque material was in the form of extremely small globules, most of which gave decided reflections when viewed in reflected light and are probably of a metallic nature.

The alumina crystals were present to an amount not exceeding a few per cents and are very small. Owing to their minuteness and the fact that they were intimately associated with the sillimanite crystals, their identity was established only through a study of immersed grains since their presence was not apparent in the thin section.

Conclusion.—Owing to the lack of information given it is impossible to discuss and compare the products examined, other than to state that with one exception the chief constituent in

every case was crystallized sillimanite, that this constituent agreed optically with the constants found for it in previous investigations and that the development as well as the size of crystals was much coarser than that normally found in porcelain and in ordinary refractory clay bodies.

Conclusions

1. It would be impractically, if not impossible, to convert fire clay into sillimanite by the methods employed in the manufacture of silica and magnesite brick.

2. The microscopic and petrographic examination of the sillimanite developed by my process leads to conclusion that the sillimanite developed by my process, free from any flux, is a stable compound and in comparatively larger crystals than any sillimanite crystals yet produced.

3. No mineral particles remain unaltered. This produces a compound of great resistance against chemical action and thermic changes, whereas in a compound containing unaltered mineral particles continuous alterations will take place, causing spalling and deterioration of the refractories.

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especially to Mr. Klein, who by tedious work helped to solve the question of the presence of corundum with the fused sillimanite.

The author also wishes to express his indebtedness to Mr. C. W. Hathaway, chief metallurgist of the National Enameling & Stamping Company, for permission to use the laboratory, microscope and the petrographic camera of that company, and also for his help and suggestions. Also to Mr. J. V. Hartman, chief chemist of the same firm, who checked the analyses, and also confirmed the presence of copper in the iron. And for his help, with that of Mr. H. C. Deterding, in the preparation of the microphotographs of the samples of brick and of the samples after the hydrofluoric acid test.

I am also greatly indebted to Mr. J. H. Campbell, chief chemist and chemical engineer for Robert W. Hunt & Company, Chicago, for his aid, suggestions and interest for years in this work, for his good advice, and for checking some of my analyses. Also to Professor J. W. Richards for his valuable advice and corrections.

SILICIOUS SAGGER MIXTURES[1]

By M. R. Hornung

Introductory

The object of this work was the study of mixtures of clay and free silica in the form of chert and gannister (quartzite) for the purpose of increasing the resistance to load conditions at kiln temperatures. The particular problem was the construction of a mixture for saggers carrying heavy loads and fired to a temperature corresponding to cone 16.

The gannister was obtained from the Hays Station plant of the Harbison-Walker Refractories Co. and represented the regular type used in the manufacture of silica brick. It came from the Mt. Union, Pennsylvania, district. Its mineralogical character has been described in detail by Ross and Insley and Klein.[2]

The chert was secured through the kindness of Mr. Wilber Stout, Ohio Geological Survey, and Mt. T. G. McDougal, Flint, Mich., and was that found at Flint Ridge, near Zanesville, Ohio. It possessed the characteristic crypto-crystalline structure with varying amounts of quartz. The chert of coarser structure was higher in recognizable quartz grains, while that showing a finer texture consisted largely of crypto-crystalline material.

In order to make the results of as general application as possible three types of bond clay were used, namely, the Tennessee and Kentucky ball clays and the bond clay from near Enid, Mississippi. In addition Georgia and Florida kaolin were introduced in some of the mixtures and later calcined Pennsylvania flint clay.

Preparation of Mixtures

The preparation of the bodies was begun by intimately mixing the constitiuents in the dry state, the clays having been ground to pass a 20-mesh sieve. The pugging of the batches, which weighed fifty pounds each, was done in a kneading machine made

[1] By permission of the Director, Bureau of Standards.

[2] Bureau of Standards, *Tech. Paper* 116 and 124.

by the J. H. Day Co. The batches were then thoroughly wedged and from each mixture two pots were jiggered, which were 10 inches in diameter and 8 inches high with walls 1 inch thick. The dried saggers were fired to cone 13 in 48 hours and held at the maximum temperature for one hour.

Tests

It was at first planned to make the load test the principal feature of examination, but it developed that the critical point involved was not resistance to pressure but one of resistance to heating and cooling. Hence a load test was made only after a body had behaved satisfactorily with reference to heating and cooling.

The load test was conducted in the usual manner as for fire brick, but the pressure adopted was 40 pounds per square inch and the maximum temperature 1375° C., held for one hour.

The heating and cooling test consisted in stacking the saggers or pots in a bung, applying a load of 200 pounds upon the top specimen and heating the bungs. The temperature was raised at the rate of 100° C. per hour to 1200° C., and the cooling took place at the rate of 200° C. per hour for the first four hours and from there on at a much slower rate, as in the natural cooling of a kiln. A fusion test was made on a few mixtures only.

The field covered in this work consisted of three series of bodies. Each series was divided into two parts, A and B, indicating the addition of chert and gannister, respectively.

Series I–A

The compositions of the members of this series are given in table 1.

TABLE 1

No.	Chert. Per cent	Tenn. ball clay. Per cent	Ky. ball clay. Per cent	Miss. bond clay. Per cent	Ga. kaolin. Per cent	Fla. kaolin. Per cent	Chert 20–40 mesh. Per cent	Chert 40–80 mesh. Per cent	Chert through 80 mesh. Per cent
1	80	6.66	6.66	6.66	...	...	32	12.00	36.00
2	75	8.33	8.33	8.33	...	...	30	11.25	33.75
3	70	10.00	10.00	10.00	...	...	38	10.50	31.50
4	65	11.66	11.66	11.66	...	...	26	9.75	29.25
5	60	13.33	13.33	13.33	...	...	24	9.00	27.00.
6	55	7.50	7.50	7.50	11.25	11.25	22	8.25	24.75
7	50	8.33	8.33	8.33	12.50	12.50	20	7.50	22.50

As shown in table 1 the chert was ground and screened to three sizes, *viz.*, 20–40 mesh, 40–80, and dust passing the 80-mesh sieve. It was desired to produce the densest body possible, since for the particular work in view the saggers were to be subjected to heavy loads. According to Kirkpatrick[1] the combination

20–40-mesh size	40 per cent
40–80-mesh size	15 per cent
Through 80-mesh size	45 per cent

should yield this condition.

The pots made from the compositions of series 1-A had a very good appearance when they came from the kiln. They possessed a good ring and were entirely free from surface cracks. Their fire expansion or contraction, measured from a line six inches long drawn on the dry pots, was found to be as follows:

No.	1	2	3	4	5	6	7
Expansion or contraction, per cent	+3.12	+1.95	+1.82	+1.04	+0.65	—0.52	—1.56

It will be observed that a neutral condition, that is, neither expansion nor contraction, was found to prevail with between 60 and 50 per cent of chert. Of these mixtures 3 and 5 were subjected to the load test in which a pressure of 40 pounds per square inch, resulting in a total load of 1108 pounds, was applied upon the pots. Specimen 3 was badly fractured, evidently not due to the load effect but to the rapid cooling while under constraint. Specimen 5 was found to be cracked but not nearly as badly as 3 and showed a linear contraction of 0.21 per cent.

At this point the load tests were temporarily abandoned and the heating test, described above, applied. All of the pots were found to be cracked and those with the largest chert content showed the greatest amount of damage.

Series I–B

The mixtures of series 1-B were exact duplicates of series 1-A, except that ground gannister was substituted for the chert. The same sizing of the silica material was maintained. Upon firing the specimens the mixtures 1 to 5, inclusive, were found to

[1] Bureau of Standards, *Tech. Paper*, 104.

be cracked, which made the heating and cooling test unnecessary. The pots of mixtures 6 and 7 were observed to crack when fired the second time along with some pots of the second series. The expansion or contraction of series 1-B was found to be as follows:

No.	1	2	3	4	5	6	7
Expansion or contraction, per cent.....	+0.92	+0.52	+0.13	0	—0.65	—1.82	—2.86

It will be noted that the expansion was appreciably smaller with gannister, and that neutral conditions prevailed in the mixture containing 65 per cent of quartzite. This shows clearly, as was to be expected from previous information, that the rate of inversion of the quartzite is slower than that for the chert.

The results thus obtained made it plain that the resistance to heating and cooling conditions was an inverse function of the total content of free silica. It was likewise evident that chert caused the bodies to be more resistant to rapid temperature changes than the gannister, though probably the mechanical strength induced by the latter is greater.

Series II–A

It was apparent from the preceding work that the amount of siliceous material had to be decreased; and it was decided to replace it in two ways; first, by means of calcined flint clay and, second, by a siliceous grog derived from mixture 7, in series 1-A, ground to pass a 20-mesh sieve but not separated further. The compositions of this series are given in table 2.

TABLE 2.

No.	Miss. bond clay. Per cent	Ky. ball clay. Per cent	Tenn. ball clay. Per cent	Fla. kaolin. Per cent	Ga. kaolin. Per cent	Chert 20–40-mesh. Per cent	Chert 40–80-mesh. Per cent	Chert through 80-mesh. Per cent	Calc. flint clay 20–40-mesh. Per cent	Calc. flint clay 40–80-mesh. Per cent	Calc. flint clay through 80. Per cent	Calc. chert body IA–7 through 20 mesh
1..	8.33	8.33	8.33	12.5	12.5	12	4.5	13.5	8	3	9	...
2..	8.33	8.33	8.33	12.5	12.5	10	3.75	11.25	10	3.75	11.25	..
3..	8.33	8.33	8.33	12.5	12.5	10	3.75	11.25	..	....	...	25
4..	8.33	8.33	8.33	12.5	12.5	....	....	...	..	....	...	50
5..	8.33	8.33	8.33	12.5	12.5	....	....	...	10	3.75	11.25	25
									calc. flint clay, 8-20 mesh			
6..	8.33	8.33	8.33	12.5	12.5	12.5	12.5	...	..	25	...	..

It will be noted that the combination of sizes of the chert and calcined flint clay which was previously fired to cone 13 is the same as for series 1.

These pots came from the kiln in very good shape but all cracked in the heating and cooling tests. The cracks were very fine and in some cases had to be looked for very carefully, being almost hair lines. Body 6 had only a very minute crack and was probably the best mixture up to this point.

Series II–B

This series was an exact duplicate of series 11-A except that gannister and the calcined gannister body IB-7 were substituted for chert and calcined chert body.

These specimens came from the kiln in very good shape but failed in the heating and cooling test, showing larger cracks than the pots of series 11-A.

The fire shrinkage of the two series, 11-A and 11-B, were as follows:

II–A No.	1	2	3	4	5
Contraction, per cent	3.1	3.7	1.8	2.5	3.7
II–B No.	1	2	3	4	5
Contraction, per cent	4.1	4.4	2.5	3.4	4.4

In this series, while there was an improvement in the resisting quality of the pots subjected to the heating and cooling tests, it is noted that even in bodies 5-A and 5-B, where the previously calcined siliceous material is reduced to 25 per cent, failure is met with, due perhaps to lack of fineness. Here again the gannister series shows cracking to a greater extent.

Series III

In this series it was decided to use a different combination of sizes for non-plastic materials and to replace the raw by calcined chert and gannister, previously fired to cone 14. It was found that the chert, although much easier to grind in the calcined state, still retained much of its former hardness. On the other hand, the calcined quartzite could be readily broken down into its individual grains, which passed the 40-mesh sieve. No attempt was made, therefore, to prepare this material in the coarser sizes

nor to separate it into definite proportions of the smaller sizes. The clay content and proportions remained the same as in series II.

Series III–A

The compositions of these mixtures are given in table 3.

TABLE 3

No.	Clay, in proportion of series II. Per cent	Calcined flint clay 8–20-mesh. Per cent	Calcined flint clay through 20-mesh. Per cent	Calcined chert 8–20 mesh. Per cent	Calcined chert through 20-mesh. Per cent	Calcined chert through 40-mesh. Per cent
1	50	25	....	25	....	..
2	50	25	....	....	....	25
3	50	25	....	....	25	..
4	50	12.5	12.5	12.5	12.5	..

The pots made from the mixtures of this series came from the kiln in satisfactory condition but appeared to be "punky" and did not ring when struck, with the exception of 2. All of these specimens stood the heating and cooling test, but since a compact and strong structure was desired the composition of 2 was taken as a basis for further work.

The firing shrinkage of the bodies of series III-A were found to be as follows:

No.	1	2	3	4
Shrinkage, per cent	1.6	2.2	2.5	2.2

The use of gannister in the compositions of series III was restricted to one body of the composition:

	Per cent
Clay (as in series II)	50
Calcined flint clay, 8–20-mesh	25
Calcined gannister, through 40-mesh	25

This pot stood the heating and cooling test satisfactorily and had a good appearance. In the load test, maintaining a pressure of 50 pounds per square inch and a maximum temperature of 1375° C., held for one hour, the pot fractured. The fusion test showed a softening point of cone 31.

It appears from the results obtained for series III that it is possible to use a moderate amount of chert by opening up the

body through the use of coarser grog. The introduction of fine siliceous material as in mixture 2 produces a body having a good ring and which stands the heating and cooling test. Since the one gannister mixture of this series in which fine grained calcined quartzite was used also stood the heating and cooling test, it seems necessary to conclude that in every case the silcious material must be fine, especially if a dense body containing larger amounts of siliceous material is to be produced.

The present work was concluded with the preparation of two bodies, *a* and *b*, consisting of a mixture of raw clay and calcined flint clay with calcined chert and gannister, respectively, which had the following compositions.

Body.	Clay as in series II.	Calcined flint clay, 8–20-mesh.	Calcined chert through 40-mesh.	Calcined gannister through 40-mesh.
	Per cent	Per cent	Per cent	Per cent
a	50	10	40	..
b	50	10	..	40

These pots when fired had a very good appearance, especially specimen *a*, which was entirely free from surface cracks and had a better ring than *b*. The linear fire shrinkage of the chert body was 1.25 per cent and that of the gannister body 2.2 per cent. In the heating and cooling test specimen *b* cracked while *a* did not.

Specimen *a* was subjected to the load test, in which the contraction was found to be 0.6 per cent in terms of the original height. It was found to show a crack, undoubtedly due to the severe treatment, consisting in rapid heating and cooling while under constraint.

These two bodies again show the advantage of chert over quartzite and the use of finer over coarser siliceous material. The tendency of composition *a* to crack in the load test should have led to the use of a smaller amount of fine chert and a larger of calcined flint clay, preferably finer in grain than that used. It should be expected that a mixture consisting of 50 per cent of raw clay, 20 per cent of calcined flint clay and 30 per cent of chert, through 40-mesh, raw or calcined, would give very good results in every respect. Time did not permit further work.

In addition to the tests already mentioned some of the pots from series I and II were re-fired a number of times. Thus

body 6 of series 1-A and 1, 2, 3, 4 and 5 of series 11-A were all sound when re-fired six times in the regular burns conducted in the work. Mixture 4, series 11-B (quartzite), cracked in the second firing but 1 and 2, series 11-B, were still sound after two firings.

Conclusions

It is evident that no sweeping conclusions can be drawn from the limited number of mixtures and specimens owing to the variables which creep into investigations of this kind, but it would seem that the following statements are warranted:

1. The use of chert yields more satisfactory results than that of quartzite.

2. Finer ground siliceous materials are more advantageous than coarser ones.

3. Previous calcination of the chert or quartzite improves the resulting bodies with reference to heating and cooling treatment.

4. The introduction of calcined flint clay improves the general character of such siliceous refractories and counteracts their tendency to spall.

5. A neutral condition of no shrinkage or expansion in firing requires the use of between 60–65 per cent of raw chert or quartzite.

6. It appears that the use of siliceous refractories for saggers and other purposes, especially with the addition of chert, is entirely feasible and that the resulting products can be made to combine excellent resistance to load conditions and permanence of volume with satisfactory behavior under conditions of heating and cooling. A mixture of 50 per cent of raw clays, corresponding approximately to the proportions used in this work, 20 per cent of calcined flint clay and 30 per cent of chert, raw or calcined and passing a 40-mesh sieve, is suggested. For each particular service there must be a definite limit for the addition of free silica which may be higher than that indicated in the present work, which covers a condition of severe use.

PITTSBURGH LABORATORY
BUREAU OF STANDARDS

CERAMIC ABSTRACTS

General

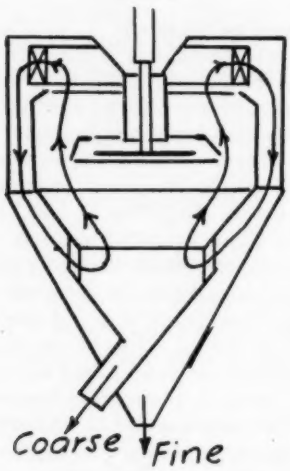
How a ceramic kiln may be insulated. P. A. Boech. *Brick Clay Record*, **55**, 1044–1047 (1919).—Ideal insulating material should possess the following properties: (1) Sufficient mechanical strength to resist wall strains due to expansion and contraction; (2) extremely high insulating value; (3) should not be decomposed or altered by high temperatures; (4) should be sufficiently refractory so as not to fuse or shrink when subjected to high heat, having approximately the same expansion as the brickwork; (5) light weight; (6) convenient form to be readily applied by unskilled labor; (7) low cost. The insulating material Sil-O-Cel is 25–30 lbs. per cubic foot naturally, or 8 lbs. per cubic foot for the loosely packed material. This material has best insulating properties when tamped to a density of about 15 lbs. per cubic foot. The melting point is 2930° F. (1610° C.). The internal conductivity varies between 0.5 and 0.8 B. t. u. per sq. ft. per hr. per degree Fahrenheit difference in temperature between ordinary temperature and 1800° F., being one-tenth that of high-grade fire brick between these temperatures. The insulating brick weigh about $1^3/_4$ lbs. each and have a crushing strength of over 400 lbs. per sq. in. For temperatures in excess of 1800–2000° F. it is desirable to use an insulating brick which has been kiln-burned to give it greater permanency in volume. These brick have a crushing strength of about 1200 lbs. per sq. in. but do not possess as good insulating value as the regular brick. Crowns can be best insulated with either a $2^1/_2$ or $4^1/_2$ inch course of insulating brick laid in a special mortar but not bonded to the brickwork of the crown. When kilns are unprotected they should be covered with one inch of insulating cement and then properly waterproofed with asphalt or another layer of brick. Insulator powder to a depth of 4–6 inches may be used in place of the brick, and this is then covered with an insulator cement. The side walls should contain a $4^1/_2$ inch thickness of the insulating brick, laid directly behind the 9-inch course of fire brick and the exterior brick. Considerable heat is lost through the base and foundation of the kiln. This can be reduced to a minimum by installing a thickness of 4 to 6 inches of insulating concrete directly underneath the fire brick bottom. The use of insulation has cut down radiation 50–75 per cent. The coal saved per year in a 30-foot kiln amounts to $230 per year in some cases while the insulation costs only one-third of this.

H. G. Schurecht.

Air separator. *Keram. Rundschau*, **27**, 295–296 (1919).—An air separator manufactured by the Pfeiffer Co., Germany, for 30 years was found to be an excellent substitute for screens and occupies only a small space. In this

separator no dust room is necessary for settling the dust as this takes place in the outer partition of the separator. The material is fed in the hopper (see diagram) from which it is thrown to the sides of the separator. Here it meets the upward current of air which takes the fine dust and tosses it to the

Air separator



outer partition as the arrows in diagram indicate. Here the fine dust settles and the air returns to the inner chamber again, thus further separating the dust from the settling coarser material. For the extremely fine separation the dust from this first separator is run through another which has a series of plates between the distributor and the air current, which affect a more thorough separation. H. G. SCHURECHT.

Patents

Tile-press. H. STOMMEL. U. S. 1,321,160, November 11, 1919. This is a power-operated dry-press. The pressing head is screw-operated, power being applied through vertical friction discs which operate either to raise or lower the head by contacting on opposite sides with the usual horizontally rotating flywheel. Leaf springs operate to check the upward movement of the head without jar. The bottom number of the die is fixed upon the usual table, the sides being movable relative thereto. In operation these latter members are raised and held in proper position by a spring. The material to be pressed is fed to the die in the usual manner and the pressing head lowered. When the head contacts with the side member of the die the supporting spring is released and the sides descend with the head. In order to discharge, the side member is lowered below the bottom.

Briqueting-press. W. SCHUMACHER. U. S. 1,322,960, November 25, 1919. A power-operated lever press in which the sides of the die are slidable and the counterdie resiliently mounted.

Utilization of greensand. E. HART. U. S. 1,322,900, November 25, 1919. Greensand consists of ordinary silica sand coated with thin layers of glauconite. By tumbling the greensand in a tumbling barrel this glauconite can be removed and collected. Potassium alum can be formed by treating the glauconite with H_2SO_4, about 66° Bé., and allowing to stand, the acid reacting with the major portion of the iron, aluminium and potassium to form sulphates, heating to about 600° C. in a muffle, quenching the heated mass in water, keeping the mass wet for several weeks and lixiviating with hot water.

Treatment of greensand, marl and glauconite. E. HART. U. S. 1,323,228, November 25, 1919. The metallic oxides present are converted into chlorides in solution; an oxidizing agent added to convert ferrous chloride to ferric; the solution evaporated to dryness and heated to between 300° and 500° C. and then treated with steam to convert ferric chloride into oxide with liberation of HCl. The soluble chlorides remaining are dissolved and calcium and aluminium salts removed, leaving potassium chloride in solution. This may be concentrated and used as a crude fertilizer or in the preparation of pure compounds of potassium.

Method of treating potassium-bearing silicates. W. GLAESER. U. S. 1,323,464, December 2, 1919. A method treating potassium-bearing silicates to form water-soluble potassium salts. The silicates are heated at a low red heat, suddenly cooled, intimately mixed with pyrites and calcium sulphate, briquetted and exposed to air and moisture to oxidize the pyrites to ferric sulphate and then burned at above 900° C. in the presence of air and steam.

Recovery of potassium and aluminum compounds from greensand. M. HAUBER. U. S. 1,323,764, December 2, 1919. The greensand is intimately mixed with ferrous sulphate and heated at a dull red heat, thereby decomposing the ferrous sulphate and forming combined sulphates of potassium and aluminium.

Sound-record and process for making it. R. HEAD. U. S. 1,321,912, November 18, 1919. Fine modeling clay is substituted for the wax-like materials usually used in sound recording. When the record is made the clay is dry so that the groove made by the recording stylus may retain its exact configuration. After the record is made it is fired as pottery is fired. A metal matrix is made from this and the final records made as usual. The inventor states that instead of clay he may use plaster of Paris or a cement of a consistency and condition just about to set, the sound groove being formed just prior to the setting or hardening of the material.

Heat-conserving composition. M. EDLUND. U. S. 1,322,098, November 18, 1919. A composition consisting of clay, sawdust, cork, hair and shoddy.

G. E. MIDDLETON.

Refractories

The reversible expansion of refractory materials. J. W. COBB and H. J. HODSMAN. *Pottery Gaz.*, **501**, 1348–50 (1919).—The expansion of calcined alumina and silica bars were measured for comparison. For the alumina bar the expansion was found quite uniform with an increase in temperature, but for silica it was found to increase suddenly between 500°–600° C., due to a transformation of α-quartz into the β-quartz, and above this temperature it had practically no coefficient of expansion, and there was no inversion to trydimite. Tests on a clay showed a curve between that of alumina and silica with quite a rapid change in expansion in the neighborhood of 500° C., due to the silica in the clay. The coefficient of expansion of carborundum was very similar to that of fused alundum, while the expansion of magnesite was very high. H. G. SCHURECHT.

Nova Scotia fire clays make good refractories. *Brick Clay Record*, **55**, 1037–38 (1919).—The fire clay described is on the island of Cape Breton, Nova Scotia. Two clays, one from Truro, N. S., and another from Sydney, N. S., are mixed together and make good refractories for coke oven walls, gas producer linings, and so forth. The chemical analyses are as follows:

	1	2
Organic matter	7.76	4.46
Silica	71.48	79.56
Alumina	17.91	13.87
Ferric oxide	1.30	2.23
Lime	.55	Trace
Magnesia	Trace	Trace

1. From Truro, N. S., and has a fusion point of 1670° C.
2. From Sydney, N. S., and has a fusion point of 1750° C.

H. G. SCHURECHT.

Some comparative tests of machine-made and hand-made silica brick. L. BRADSHAW and W. EMERY. *Pottery Gaz.*, **501**, 1338–39 (1919).—The conclusions arrived at by the authors are as follows: (1) The prevailing objections to machine-made silica brick appeared to be due to prejudice arising from early failures, due to improper mixing and using unsuitable machines; (2) in recent years the quality of machine-made silica brick has been greatly improved; (3) in no tests were the machine-made brick found inferior to the hand-made brick while in several important respects, *viz.*, the mechanical strength, resistance to spalling and abrasion and in accuracy of shape, they are superior to the hand-made brick. H. G. SCHURECHT.

Specific heat of refractory materials at high temperatures. L. BRADSHAW and W. EMERY. *Pottery Gaz.*, **510**, 1338 (1919).—The specific heats of a number of refractories were determined at temperatures ranging from 600°–

1400° C. The method was to heat a weighed test piece of the material to the required temperature and then quickly drop it into a known weight of water and to measure the rise in temperature produced. Heating was done in an electric furnace placed vertically over the tank, which was surrounded by insulating material and enclosed in a wooden case. The results show that the specific heats of materials increased at high temperatures, the increase in the case of fire clay and silica brick between 100° and 1400° C. being approximately 50 per cent. This means that the amount of heat required to raise the temperature of a given weight of material through an interval of 100° C. from 1200° to 1300° C. was greater than that required to raise the temperature from 100° to 200° C. H. G. SCHURECHT.

Refractories in the zinc industry. J. A. AUDLEY. *Trans. Ceram. Soc.* (Eng.), **18**, 43–66 (1919).—The retorts or muffles are made of a mixture of raw fire clay and burned clay or clean fragments of old retorts; the mixture is more resistant to fluxes the higher the ratio of alumina to silica, and accordingly the addition of coarse-grained quartz (as in Upper Silesia) to the retort mixture has not proved successful with basic charges. The great objection to the ordinary mixtures is that clay is a bad conductor of heat, and will not stand great fluctuations in temperature, that it is attacked by material in the charge, and that its porosity allows zinc vapor to escape. The chief requirements of a zinc retort include refractoriness, resistance to dust and slag, mechanical strength and tenacity, resistance to sudden temperature changes, low degree of permeability to zinc vapors, minimum contraction in the fire, and durability. In practice great influence is exercised by the process of preparing the mixture. In Silesia the mixture is composed of two volumes of raw rich clay (Briesen, Saarau, or Striegau, or mixtures of these), one of raw sandy clay (Striegau clay, or Silesian kaolin, or mixtures), and five of other materials. The latter are powdered fragments of old retorts and burned Neurode shale, mixed in different proportions. The size of the clay grains is from $^1/_{25}$ to $^3/_{25}$ inch in diameter. The larger pieces increase the strength of the muffle and make it more resistant to temperature changes and chemical erosion. In Belgium the proportions vary much. In some works 40 volumes of rich raw clay, 50 of burned clay, and 10 of coke are used. In other works the corresponding figures are 36, 54, and 10, respectively, and in still other cases the proportions are 30 of rich raw clay, 10 of sandy clay, 50 of retort fragments, and 10 of coke. Another Belgian composition is 10 of raw clay, 10 of burned clay, 8 of retort fragments, one of quartz, and 0.5 of coke. Coke added to the mixture for making retorts or muffles, improves the refractory qualities of clay and reduces shrinkage. In Belgium and Westphalia the retorts are often glazed externally in order to render their sides impervious to zinc vapor. In many Belgian works, and in Spain, much quartz or sand is added to highly siliceous clay, so that the retorts consist chiefly of silica, which stands fire well, is a good conductor of heat, and enables thinner vessels to be made, but it is certainly less resistant than alumina to fluxes, though not so much corroded by

siliceous ores. These retorts are often also glazed. The condensers are made of less refractory materials than the distillation vessels. In many places they are made of equal parts of raw and burned clay, or of ordinary clay mixed with ground retort fragments and coke or coal ash.

The walls of new retorts absorb zinc at first, forming zinc aluminate (artificial zinc spinel), which gives the well-known blue color. The average zinc content of old retorts is 6 per cent, but may rise to as much as 21 per cent, as at the Bethlehem Zinc Works, Pennsylvania; in seven samples of broken retorts from Upper Silesia the average amount of metallic zinc was 12.87 per cent. The addition of coke to the retort material, and the employment of heavy pressure in making the retorts, has resulted in greatly reducing the absorption of zinc. The fine dust from the coal (consisting of silica, clay substance, iron oxide or pyrites, lime, magnesia and alkalies), attacks the clay substance of the porous vessels, forming a gradually thickening glaze. The zinc vapors in the meantime will change the clay substance into zinc aluminate (zinc spinel), the silica—both that from the clay substance and quartz grains in the clay—being partly transformed into tridymite. The zinc spinel is formed particularly during the first time of using the vessels, while zinc vapors and combustion gases can diffuse readily through the retort before the coating of glaze is formed. Thus, after being in use a few days, the retorts do not consist of fire clay, but mostly of zinc spinel and tridymite. A zinc retort is thus a porous vessel, more or less damaged by various cracks, mainly consisting of clay substance, zinc spinel, and quartz or tridymite, with modifications in parts through local vitrification. In the zinc furnace the vessel can resist powerful mechanical influences, because the fired body, though brittle when cold, has great tenacity in the white hot state. The degree of impermeability to zinc vapors depends largely on the kind and number of external and internal surface cracks, and on the density and viscosity of the glaze. The use of magnesia for retorts has proved unsatisfactory. Coatings of graphite and clay and of fused bauxite and zirconia are suggested.

A. J. LAMBERT.

Patents

Refractory cement. H. B. BOOTH. U. S. 1,321,085, November 11, 1919. This cement is designed particularly for protectively coating electrodes. It consists of feldspar 15–40 per cent, artificially crushed quartz 40–20 per cent, silica sand 15 per cent, and 30–15 per cent of a binder consisting of sodium silicate 92 parts, sodium sulphate 8 parts, and water. The feldspar content is varied, depending upon the heat. For an electrode heating to 3000° F., 15 per cent feldspar, 40 per cent crushed quartz is used, while for an electrode heating to 1500° F. 40 per cent feldspar, 20 per cent crushed quartz is preferred.

G. E. MIDDLETON.

Enamels

Materials and methods used in the manufacture of enameled cast iron wares. HOMER F. STALEY. U. S. Bur. of Standards *Tech. Paper* **142.**—This

is a comprenehsive treatise dealing with the various phases of the technology of manufacturing enameled cast iron wares. It is the first publication dealing fully with this subject in any language.

The paper deals particularly with the coating of cast iron with white vitreous enamels by the American or dry-powder process. In this method of manufacture, the thoroughly cleaned castings are painted with a suspension in water of powdered glass, clay and other materials. This suspension is known as the ground coat. The castings are then heated until the ground coat is thoroughly fused, forming an extremely thin layer of transparent glass on the castings. The ware is then withdrawn from the furnace, and immediately coated by means of long-handled sieves with a layer of white, opaque, powdered glass, known as the cover coat enamel. Next the castings are returned to the furnace, and in a couple of minutes the cover coat enamal fuses to a smooth layer. Two such layers of cover coat enamel are usually applied, giving a total thickness of from $^1/_{32}$ to $^1/_{16}$ of an inch. The paper does not discuss the enemaling of cast iron with wet coat cover enamels.

Several sections are devoted to a discussion of the preparation and testing of the various chemicals and minerals used in compounding enamels and to the effect each of these has on the physical and chemical properties of enamels. The methods emloyed in making and cleaning the castings are described in detail, beginning with construction of patterns and ending with sand blasting and polishing. The methods and equipment, including furnaces, used in preparing enamels and applying them to castings are covered in two sections.

Sections are devoted to the control of the luster of enamels, the cause and control of crazing, and to the methods to be used in avoiding blisters, pin holes and other minor defects. Typical formulas are given for ground coat enamels, cover coat enamels containing oxide of tin, and for those containing antimony compounds as the opacifying agent. These formulas are each stated in terms of raw batch for 1000 pounds melted enamel, of percentage composition and of empirical chemical formula. The calculations involved in arriving at each form of expression are explained in detail. R. R. DANIELSON.

Fuel consumption of muffle kilns in the enameling industry. L. VIELHABER. *Keram. Rundschau*, **27**, 293–294 (1919).—The question whether producer-gas fired kilns are better than semi-gas fired kilns in the enameling industry is important to those installing new kilns. It is admitted that the producer-gas ovens are successful in many metallurgical industries and will be important for the larger enamelware companies, but it is doubtful whether they are much better than the semi-gas fired kilns now used extensively in Germany for small plants. A heat balance was run on an enamel muffle-oven which in 24 hours burns 1800 kg. of ware and 500 kg. of coal. The calculations were as follows:

Specific heat of enamel and iron = 0.15.
Melting point of enamel = 950° C.
Heat used in heating enamel and iron = 0.15 × 1800 × 950 ..... 256,500 heat units
Heat of fusion of enamel = 70 heat units.
540 kg. of the 1800 kg. of ware is enamel, hence heat used in melting enamel = 70 × 540 ..... 37,800 heat units

Heat used ..... 294,300 heat units
Heat lost through opening of doors = 24 × 15 × 2640 ..... 950,000 heat units

Since doors are opened 15 times per hour, through which the temperature drops 30° C.

The muffle has a volume of 0.224 cbm. of fire brick which weigh about 400 kg.

The specific heat of the brick is 0.22 and hence to heat 400 kg. to 30° C., 0.22 × 30 × 400 = 2640 heat units are used.

The producer gas is heated to 450° C. One kg. of coal produces 12.8 cbm. of gas, which has a value of 6592 heat units. In 24 hours 500 × 6592 = 3,296,000 heat units are given up by the gas.

Theoretical air necessary = 6.04 kg.
Practical air necessary = 8.45 kg.
Excess air = 2.41 kg.
Theoretical products of combustion = 11.45 kg.
Products of combustion from 1 kg. coal = 13.86 kg. or 6930 kg. for 500 kg.

Since gases leave recuperator at 500° C., the following heat is lost in the smoke-stack: 0.255 × 500 × 6930 = 883,575 heat units.

Heating enamel	256,500 heat units
Melting enamel	37,800 heat units
Radiation loss	1,186,560 heat units
Lost through opening doors	950,000 heat units
Lost through chimney	883,575 heat units
	3,314,435 heat units

If the gas passes to recuperator at 800° C. we get:

0.255 × 800 × 6930	1,413,720 heat units
Heat passing through chimney	883,775 heat units
Saved by recuperator	529,945 heat units

If air in recuperator is heated to 490° C., 520,240 heat units are saved by the recuperator, or 76 kg. of coal are saved per 24 hours.

Heat used is as follows:

Enamel heating	256,500 heat units
Heat of fusion	37,800 heat units
	294,300 heat units

Converting coal in generators to gas also requires heat, the coal having a heating value of 7530 and the gas from 1 kg. of coal 6592 heat units, so 939 heat units are used in converting 1 kg. of coal to gas, or 469,800 heat units for 500 kg. Judging from these results the author believes that the semi-gas fired kilns with a small recuperator would give results very similar to the above. H. G. SCHURECHT.

Patents

Rheostat. L. KEBLER. U. S. 1,321,104, November 11, 1919. A ground coat and a glaze are applied to the cast iron base and fired. A second ground coat and glaze are then applied upon this and the whole fired. The resistance elements are placed upon this second glaze surface, covered with a third glaze or enamel and the whole again fired. With the interposed relatively infusible ground coats there is no danger of the resistance elements sinking through the glaze and contacting with the base during the final firing.
G. E. MIDDLETON.

Whiteware and Porcelain

The gelatinization of clay-casting slip. J. W. MELLOR. *Pottery Gazz.*, **510,** 1338 (1919).—When sodium silicate was used in casting slips it was noted that the slip gelatinized or became slimy, usually on the surface. Works in some districts were troubled with this more than plants in other districts. This may be due to the action of CO_2 in the air on the slip, which tends to coagulate it. Lime in water in the form of the acid carbonate may also coagulate the slip, so experiments were made in purifying water and comparing same with the unpurified tap water. It was found that water purified by the "Permutit" process permitted one to prepare a slip with a greater weight per pint and with a less percent of soda than was the case when the impure tap water was used. H. G. SCHURECHT.

Patents

Kiln car. J. B. OWENS. U. S. 1,321,388, November 11, 1919. This car is designed particularly for use in an Owens tunnel kiln. Its frame is a unitary cast iron structure carrying a platform of fire brick. Each frame is provided with an integral projecting bumper which contacts with the adjoining car at its center. By thus applying the pushing pressure at one central point, warping of the car frames does not result in the jamming of the whole train. There is a tongue and groove connection at the adjoining ends of the frames.
G. E. MIDDLETON.

Glass

Patents

Glass-drawing tank. W. A. JONES. U. S. 1,321,752, November 11, 1919. A buoyant division wall between twin glass tanks is provided. By extending only a short distance down into the molten glass it does not interfere with a free flow thereof, but it does prevent a free sweep of the flames from one tank to the other.

Glass-drawing bait. G. C. DEBAY. U. S. 1,321,810, November 18, 1919. As the drawn cylinder is swung into horizontal position preparatory to splitting and cutting, the blowpipe is automatically released from the bait. This relieves the cylinder from the weight of the blowpipe and prevents the cracking which often results because of this weight.

Mold-loading device. R. E. MCCAULEY. U. S. 1,322,318, November 18, 1919. The glass furnace is provided with an extension into which flows the glass to be dispensed. An aperture is provided in the lower part of this extension and a tubular member connected with a vent and a suction pipe is provided in the extension above the aperture. By reducing the air pressure in the tubular member, glass is drawn upwardly therein and prevented from discharging through the aperture. The pressure is then allowed to return to normal and the column of glass descends, passes out through the aperture and into a mold positioned to receive it.

Glass-gathering and blowing machine. M. J. OWENS. U. S. 1,322,726, November 25, 1919. This invention is designed as an improvement to the "Owens" bottle machine. In that machine a rotary frame carries a series of blank molds over a pool of glass, the glass is sucked up through the bottom of the mold, the glass in the mold severed from that in the tank, the blank is blown in the blank mold, the knife is withdrawn leaving the blank suspended from the neck mold, the blow mold is then placed in position and the final blowing effected. The present machine employs a pneumatically operated plunger which serves as a support for the blank after the immediate withdrawal of the knife, as well as a bottom for both blank and blow molds. When the blank mold is removed the blank remains positioned upon this support and not suspended from the neck mold as formerly.

Glass-annealing oven. W. STENDER. U. S. 1,322,735, November 25, 1919. The annealing bed is intermittently moved through the oven. At the feed end of the oven, a semi-circular article supporting plate is supported immediately above the bed. An annular feed device provided with peripheral pockets rotates above the supporting plate and positions articles to be annealed thereupon as they are fed from the molding machine. When the supporting plate is filled, the articles are automatically swept off upon the annealing bed which then moves forward another step.

Glass-furnace. H. E. DEVAUGHN. U. S. 1,322,767, November 25, 1919. In the operation of glass-furnaces, the piers built up from the bottom or from the walls to support the cap of the furnace and to separate the drawing chambers, are frequently destroyed by the molten glass. This is obviated by suspending the skew blocks and the furnace cap over the tank or furnace. Means are provided for protecting the suspension devices from the intense heat.

Vial-necking machine. W. D. FREDRICK, W. E. GLASPEY and C. F. COX. U. S. 1,322,778, November 25, 1919. Small glass bottles or vials are made from previously formed glass tubes by cutting to proper length and then forming bottom and neck. This operation is usually performed by hand. This machine forms the neck automatically, the operation being performed simultaneously upon both ends of a blank, and from the partly finished blank so prepared two vials may be produced, such blank being separated intermediate its ends and the two sections separately bottomed by the machine shown in U. S. 1,322,779.

Vial-bottoming machine. W. D. FREDRICK and W. E. GLASPEY. U. S. 1,322,779, November 25, 1919. The double-necked blank formed in the machine shown in U. S. 1,322,778 is intensely heated at its middle, stretched to breaking and then both bottoms thus formed flattened by being pressed against an interposed double-ended anvil.

Method and apparatus for drawing wire glass. I. W. COLBURN. U. S. 1,323,389, December 2, 1919. Wire cloth passes around a roller slightly immersed in a tank of molten glass and passes off horizontally embedded in a sheet of glass. It then passes between cooling rolls.

Process and apparatus for feeding molten glass. B. D. CHAMBERLIN. U. S. 1,323,450, December 2, 1919. The glass after passing the discharge orifice passes through a heated tube of larger diameter than the glass stream and terminating in a water-cooled shearing edge. Beneath this is situated an intermittently rotating, combined receiving and shearing member. When the upper pocket of this member has received the proper quantity of glass the member is rotated through 180°, shearing the glass stream, depositing the glass charge in a mold positioned below and presenting its other pocket for the reception of further glass.

Glass-mold charging apparatus. F. L. O. WADSWORTH. U. S. 1,323,507, December 2, 1919. A stream of molten glass flows from the tank into a mold beneath the orifice. When the proper amount of glass has entered the mold, the stream is sheared by two approaching blades situated immediately beneath the orifice, forming, when in contact, a V-shaped trough which momentarily supports the glass. A two-part intermediate supporting cup is then shifted into place beneath the shearing blades which are then separated, allowing the glass to flow into the supporting cup. Meanwhile an empty mold is moved into position, the supporting cup is opened and the glass allowed to flow into the mold.

Cement and Lime

Preheating air for rotary kilns. S. H. HARRISON. *Concrete* (Cement Mill Section), **16**, 9 (1920).—It has been shown that about 11 per cent of the heat applied to a rotary kiln in the burning of Portland cement remains in the hot

clinker as it leaves the kiln. One of the most practical methods of reducing this loss is to utilize the heat of the clinker to preheat the air necessary to support the combustion of the coal in the kiln. It is necessary to control the amount of air allowed to pass through the clinker cooler into the kiln so that there will not be too great an excess of air. The clinker leaves the kiln at a temperature of about 1200° C. and gives up to the air approximately 520 B. t. u. per pound of clinker. Assuming that the theoretical amount of air for complete combustion of the coal enters the cooler at atmospheric pressure it is calculated that 480° C. is the maximum temperature to which the air is, heated. Curves are given showing the relation between the temperature of the preheated air and the weight of coal per pound of clinker, for amounts of excess air from 50 to −10 per cent. The highest theoretical temperatures are obtained when the fuel consumption is lowest. There should be a slight excess of air rather than a deficiency, in order to obtain the highest kiln efficiency.

F. A. KIRKPATRICK.

New wet process plant of the Indiana Portland Cement Co. STAFF ARTICLE. *Concrete* (Cement Mill Section), **16**, 1 (1920).—The plant is located near Greencastle, Ind. Reinforced concrete is the material used for construction of buildings, tanks, piers, and so forth. The storage pits are of the following capacity in tons: Limestone 5,600, shale 1,600, clay 1,500, clinker 41,000, gypsum 580, coal 1,400. Raw materials are hauled in $3^1/_2$-yard dump cars to the storage bins. The clay is mixed with 60 per cent of water and reduced to a slurry in a specially designed disintegrator 20 feet in diameter and 6 feet deep. The dry crushed limestone and shale are run into the clay slurry, the mixture having from 30 to 36 per cent water. The slurry is then passed over screens having openings of 0.049 inch and then ground in a tube mill until 88 per cent passes a 200-mesh sieve. It is then run into three correcting basins each 18 feet in diameter and 10 feet deep, and from these into a mixing basin of 1200 barrels capacity. It is then lifted by a compressed air lift into a storage tank above the kiln. This kiln is 10 feet in diameter and 240 feet long, lined with Louisville fire brick for 210 feet, and lifter plates fastened to the upper 30 feet stir the slurry to bring it into contact with the hot gases. The clinker passes into a rotary cooler 7 feet by 66 feet, containing lifter plates to stir up the clinker and with a specially designed fan at the lower end. The clinker drops from the cooler into storage, from where it is taken by crane as required for grinding. Reduction is accomplished in two steps, first to 65 per cent of 100-mesh size in a comminutor, then to flour in a tube mill. The fuel is powdered coal. The coal is passed through a Cummer dryer, then ground in Fuller mills. Detailed layout of the plant and a number of photographs are given.

F. A. KIRKPATRICK.

Patent

Utilization of low-grade carboniferous material in the manufacture of Portland cement. R. W. LESLEY. U. S. 1,323,294, December 2, 1919. Low-grade carboniferous materials such as oil shale, lignite and low-grade

bituminous and cannel coal are heated to drive off their volatile constituents, leaving an argillaceous residue and fixed carbon. This residue is mixed with calcareous material and charged into a rotary cement kiln. The volatile constituents first driven off as stated above, together with the fixed carbon in the residue, serve as fuel. The hot gases given off during this clinkering operation are used in the initial process of driving off the volatiles. Thus is secured a self-contained process of forming Portland cement clinker.

U. S. 1,323,293 is drawn to the apparatus used in carrying out the above process.

Process of making cement. H. D. BAYLOR. U. S. 1,323,952, December 2, 1919. Cement is made slow-setting and plastic by adding CaO thereto, comminuting the mass, adding oily or waxy material and simultaneously stirring in the presence of an amount of water sufficient to hydrate all the CaO, and finally grinding.

Cement composition. J. GAISSER. U. S. 1,322,893, November 25, 1919. A plastic composition consisting of approximately 10 parts of superfine cement, 4 parts hydrated lime, 2 parts marble dust and water.

G. E. MIDDLETON.

ACTIVITIES OF THE SOCIETY

Actions of the Board of Trustees

December 29, 1919. It was voted to print the standard clay tests proposed by the Committee on Standards and submit them to the voting members for adoption.

January 13, 1920. It was voted to appoint Mr. H. F. Staley, Editor of the JOURNAL in place of Mr. G. H. Brown, resigned.

February 2, 1920. It was voted that the American Ceramic Society become a member of the International Chemical Union, thus becoming entitled to the publications issued and the recognition accorded members of this association and that the Society authorize the annual payment of the fees of approximately the amount stated, a sum not to exceed $40 per year.

New Members Received during January

Resident Associate

Blanchard, C. R., General Electric Co., Pittsfield, Mass.
Brenner, R. F., Chief Chemist, Bartlett-Collins Glass Co., Sapulpa, Okla.
Brett, R. C., Southern Clay Mfg. Co., North Birmingham, Ala.
Burdick, Percy W., Ceramic Chemist, Carborundum Co., Niagara Falls, N. Y.
Burgess, M. L., Secretary-Treasurer, Marietta Mfg. Co., Indianapolis, Ind.
Healey, A. S., Superintendent, Standard Sanitary Pottery Co., Elizabeth, N. J.
Hibbs, Jos. S., Assistant General Manager, J. W. Paxson Co., Philadelphia Pa.
Leibson, J. S., General Electric Co., Schenectady, N. Y.
Lintz, E. H., Jewett Refrigerator Co., Lackawanna, N. Y.
Menne, L. H., Enameler, Theo. A. Kochs Co., Chicago, Ill.
Morey, Geo. W., General Manager and Scientific Director, Spencer Lens Co., Hamburg, N. Y.
Palmer, Ralph M., E. I. du Pont de Nemours & Co., Wilmington, Del.
Payne, A. R., Chief Physicist, Hazel-Atlas Glass Co., Clarksburg, W. Va.
Stone, Charles H., Jr., Research and Industrial Chemist, Kemet Laboratories Co., Cleveland, Ohio.
Wiester, Stefan, Enameler, Malleable Iron Range Co., Beaver Dam, Wis.
Wright, Harry E., Superintendent, Kokomo Opalescent Glass Co., Clarksburg, W. Va.

Foreign Associate

Musiol, Charles, 16 rue de la Bigorne, Brussels, Belgium.
Saxton, Clement, Assistant Manager, Les Verreries, Aigues Mortes (Gard), France.

$6,831.35

The Value of Fuel Saved in One Year

¶ Actual records were kept for 365 days of the burning of 9-inch, high-grade, refractory brick in one continuous tunnel kiln and in seven 30-foot round kilns, during which time 5,110,000 brick were burned in the tunnel kiln as against 5,040,000 in the seven round kilns.

¶ But this isn't all—the actual labor saving amounted to $5,808.00. Taking into consideration the necessary items of depreciation, interest on plant, maintenance and repairs, the average yearly cost for burning 1,000 brick in the continuous tunnel kiln system was $2.95 as against $6.20 in the round kilns.

¶ If you are really interested in the greater efficiency of burning, you will let us tell you more about the actual accomplishments of

The Didier-March Continuous Railroad Tunnel Kiln

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Edgar Georgia Paper Clay and Kaolin	*Edgar Brothers Co.*
Lake County Florida Clay	*Lake County Clay Co.*

One Management—Office, Metuchen, N. J.

Zwermann *Twin* Tunnel Kiln

Note Its Chief Advantages:—

First:—The first cost of this kiln compared with a single tunnel of the same capacity is considerably lower. It requires less brick, one-half of the buck stays, less space, and no side or return track.

Second:—This twin tunnel kiln allows a greater utilization of the fuel than a single tunnel kiln, as the heat from cooling ware is used for water-smoking the incoming ware.

Third:—This kiln allows an absolute and positive control of the firing zone, and in the firing zone a reducing or oxidizing condition can be maintained at will.

Fourth:—The cooling of the ware as well as the water-smoking and pre-heating in this kiln are absolutely automatic. It is impossible to get the receiving end of the tunnel too hot.

Fifth:—This kiln will save about 50% on labor and from 65% to 75% on fuel as compared with periodic kilns. Burning time is cut down by ⅓ to ½. Where saggers are used, they will last twice as long.

Carl H. Zwermann **Robinson, Ill.**

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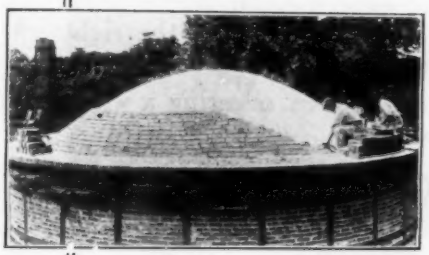

insulation is peculiarly adapted to kiln insulation as it can be applied to the portions of the kiln where greatest losses occur.

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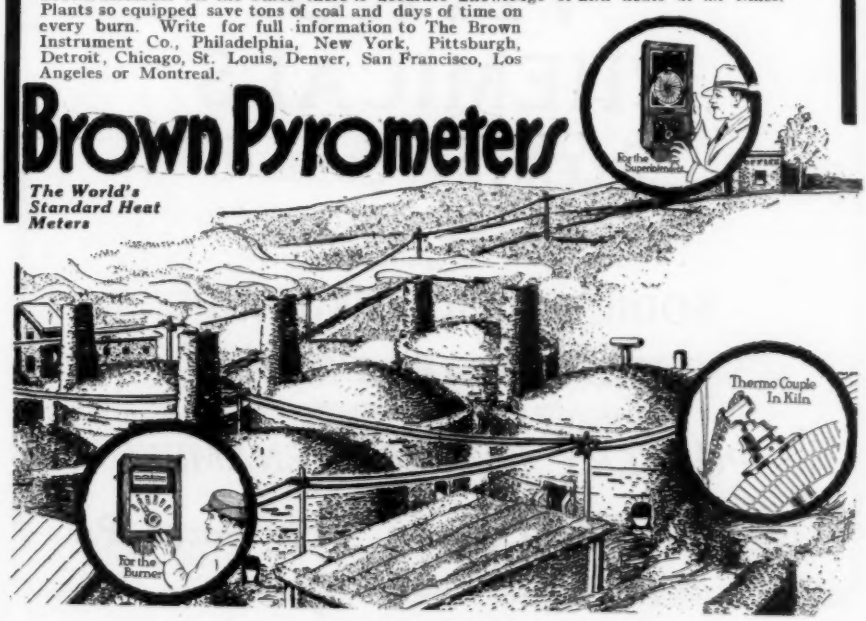

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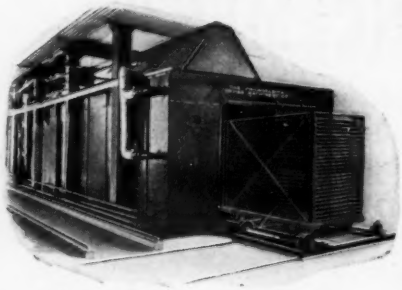

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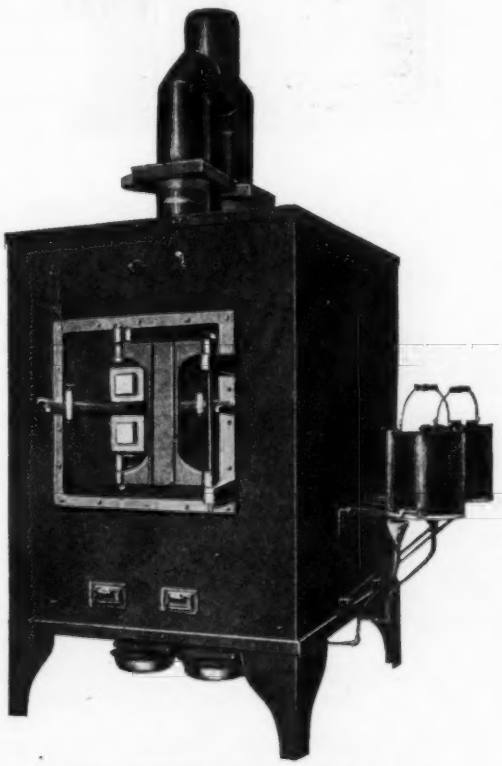

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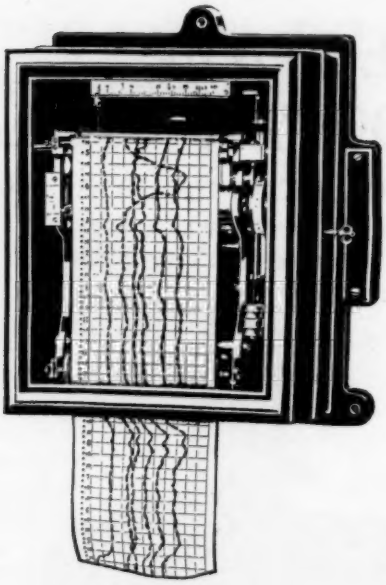

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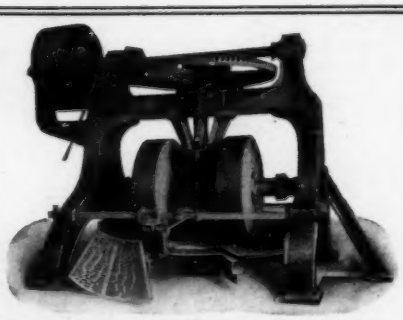

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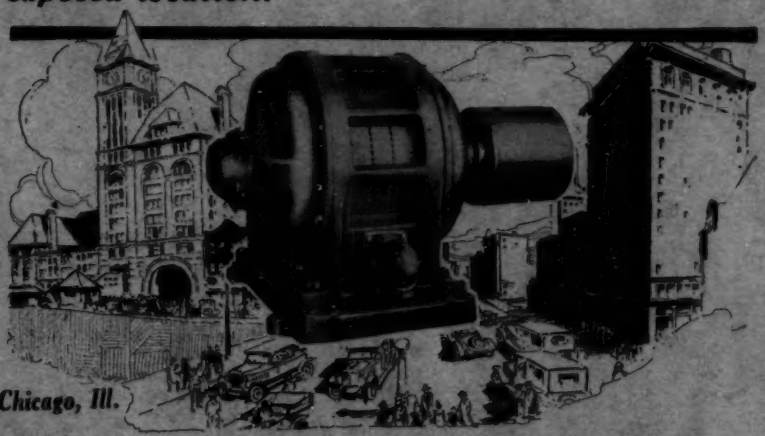

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